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# NBS SPECIAL PUBLICATION 260-105

U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Standard Reference Materials:

Summary of the Environmental Research, Analysis, and Control Standards Issued by the National Bureau of Standards

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R. Mavrodineanu and S. D. Rasberry

he National Bureau of Standards<sup>1</sup> was established by an act of Congress on March 3, 1901. The Bureau's overall goal is to strengthen and advance the nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, the Institute for Computer Sciences and Technology, and the Institute for Materials Science and Engineering.

# The National Measurement Laboratory

Provides the national system of physical and chemical measurement; coordinates the system with measurement systems of other nations and furnishes essential services leading to accurate and uniform physical and chemical measurement throughout the Nation's scientific community, industry, and commerce; provides advisory and research services to other Government agencies; conducts physical and chemical research; develops, produces, and distributes Standard Reference Materials; and provides calibration services. The Laboratory consists of the following centers:

- Basic Standards<sup>2</sup>
- Radiation Research
- Chemical Physics
- Analytical Chemistry

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- Chemical Engineering<sup>2</sup>

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Conducts research and provides measurements, data, standards, reference materials, quantitative understanding and other technical information fundamental to the processing, structure, properties and performance of materials; addresses the scientific basis for new advanced materials technologies; plans research around cross-country scientific themes such as nondestructive evaluation and phase diagram development; oversees Bureau-wide technical programs in nuclear reactor radiation research and nondestructive evaluation; and broadly disseminates generic technical information resulting from its programs. The Institute consists of the following Divisions:

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# Summary of the Environmental Research, Analysis, and Control Standards Issued by the National Bureau of Standards

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#### Preface

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards (NBS) are well-characterized materials, produced in quantity and certified for one or more physical or chemical properties. They are used to assure the accuracy and compatibility of measurements throughout the Nation. SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. They are also used extensively in the fields of environmental and clinical analysis. In many applications, traceability of quality control and measurement processes to the national measurement system is carried out through the mechanism and use of SRM's. For many of the Nation's scientists and technologists it is therefore of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values of the SRM's produced. An NBS series of papers, of which this publication is a member, called the NBS Special Publication - 260 Series, is reserved for this purpose.

The 260 Series is dedicated to the dissemination of information on different phases of the preparation, measurement, certification and use of NBS SRM's. In general, much more detail will be found in these papers than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity and accuracy of the measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing the greatest care and accuracy. These papers also should provide sufficient additional information not found on the certificate so that new applications in diverse fields not foreseen at the time the SRM was originally issued will be sought and found.

Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth, will receive prompt attention from:

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> Stanley D. Rasberry, Chief Office of Standard Reference Materials

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  - \* Send order with remittance to Superintendent of Documents, US Government Printing Office Washington, DC 20402. Remittance from foreign countries should include an additional one-fourth of the purchase price for postage.
- \*\* May be ordered from: National Technical Information Services (NTIS). Springfield Virginia 22161.

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#### Abstract

This publication is a summary of the environmental research, analysis, and control standards issued by NBS as Standard Reference Materials (SRM's). The material, composition, certification, use, and remarks concerning each of the SRM's described are presented in tabular form. Copies of the certificates of these SRM's are contained in the appendix for more detailed information.

Key Words: chemical composition; environmental standards; Standard Reference Materials.

#### Introduction

Since its inauguration in 1901, the National Bureau of Standards (NBS) has issued nearly 2000 different Standard Reference Materials (SRM's). Many of these have been renewed several times, many have been replaced or discontinued as technology changed. Today, over 900 SRM's are available, together with a large number of scientific publications related to the fundamental and applied characteristics of these materials. Each material is certified for chemical composition, chemical properties, or its physical or mechanical characteristics. Each SRM is provided with a Certificate or Certificate of Analysis that contains the essential data concerning its properties or characteristics. The SRM's currently available cover a wide range of chemical, physical, and mechanical properties, and a corresponding wide range of measurement interests in practically all aspects of fundamental and applied science. These SRM's constitute a unique and invaluable means of transferring to the user accurate data obtained at NBS, and provide essential tools that can be used to improve accuracy in practically all areas where measurements are performed.

In addition to SRM's, the National Bureau of Standards issues a variety of Research Materials (RM's) having various properties described in individual "Reports of Investigation." They are intended primarily to further the scientific or technical research on that particular material. Other materials, called Special Reference Materials (GM's), are also available from NBS. These are materials produced and certified by other Government agencies, standard organizations, or other nonprofit organizations, that are considered useful to the public and for which no alternate method of national distribution exists.

The various categories of materials available from NBS are given in table 1. This table lists these materials according to their chemical composition, physical properties, or engineering characteristics. A more detailed alphabetic enumeration of these materials is given in appendix I. Table 1 and appendix I were taken from NBS Special Publication 260, NBS Standard Reference Materials Catalog, 1984-85 Edition. This publication lists every material available from the NBS Office of Standard Reference Materials.

Further information on the reference materials available from NBS may be obtained from the Office of Standard Reference Materials, National Bureau of Standards, Gaithersburg, MD 20899. Information on other NBS services may be obtained from the Technical Information and Publications Division, National Bureau of Standards, Gaithersburg, MD 20899.

In addition to reference materials, NBS provides many additional services. These include: Measurement Assurance Programs, Calibration and Related Measurement Services, Proficiency Sample Programs, a National Voluntary Laboratory Accreditation Program, Standards Information Services, Standard Reference Data, and Technical Information and Publications.

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402, under Stock No. 003-003-02558-5 (Price \$5.50, add 25 percent for foreign orders.)

#### CERTIFIED CHEMICAL COMPOSITION STANDARDS

Steels (chip form) Gases in Metals Plain carbon High-Purity Metals Low alloy High alloy Electron Probe Microanalytical Standards Stainless Too1 Primary, Working, and Secondary Standard Chemicals Steels (granular form) Microchemical Standards Steels (solid form) Clinical Laboratory Standards Ingot iron and low alloy Special ingot irons and low alloy Biological Standards Stainless Specialty Environmental Standards High-temperature alloys Tool Analyzed gases Analyzed liquids and solids Steelmaking Alloys Permeation tubes Cast Irons (chip form) Industrial Hygiene Standards Cast Steels, White Cast Irons, Ductile Spectrochemical Standards Irons, and Blast Furnace Irons (solid form) Hydrocarbon Blends Nonferrous Alloys (chip form) Metallo-Organic Compounds Aluminum "Benchmarks" Fertilizers Cobalt Ores Copper Copper "Benchmarks" Minerals, Refractories, Glasses, and Lead Magnesium Carbides Nickel Nickel Superalloy, Trace Elements Cement Nickel oxide Trace Element Standards Selenium Tin Nuclear Materials Titanium Zinc Special nuclear materials Zirconium Plutonium assay Nonferrous Alloys (solid form) Plutonium isotopic Uranium assay Aluminum "Benchmarks" Uranium isotopic Copper Copper "Benchmarks" Neutron density standards Lead Fission track glass standards Nickel Titenium Isotopic Reference Standards Zinc

Zirconium

#### CERTIFIED PHYSICAL PROPERTY STANDARDS

Ion Activity Standards

pH standards pD standards

Ion selective electrodes

Mechanical and Metrology Standards

Magnification Coating thickness Glass Elasticity Density

Polymer Rheology

Heat Standards

Superconductive thermometric fixed point devices

Freezing Points

Defining fixed points
Determined reference points

Melting points Calorimetric

> Combustion Solution Heat source

Enthalpy and heat capacity

Vapor pressure Thermal expansion Thermocouple materials Thermal resistance

Magnetic Standards

Magnetic susceptibility Magnetic moment Paramagnetic resonance Optical Standards

Spectrophotometric Thermal emittance Refractive index

Radioactivity Standards

Alpha-particle standards
Beta-particle and gamma-ray gas
standards
Alpha-particle, beta-particle,
gamma-ray, and electron-capture
solution standards
Contemporary standard for carbon-14

dating laboratories
Environmental standards
Low energy photon sources

Low energy photon sources Gamma-ray "point-source" standards Radium gamma-ray solution standards Radium solution standards for

random analysis

Radioactivity standard reference materials currently not in stock

Metallurgical

Mossbauer

X-ray Diffraction

Gas Transmission

Permittivity

Reference Fuels

Resistivity

#### ENGINEERING TYPE STANDARDS

Standard Rubber and Rubber-Compounding Materials

Reference Magnetic Tapes

Lubricant Standards

Sizing Standards

Glass spheres for particle size Turbidimetric and fineness (cement) X-ray and Photographic Standards

Surface Flammability Standards

Semiconductor Production Standards

Water Vapor Permeance

Tape Adhesion Testing Standards

Color Standards

SPECIAL REFERENCE MATERIALS

RESEARCH MATERIALS

This work is the fourth in a series of NBS Special Publications dedicated to the description of the Standard Reference Materials (SRM's) issued by the National Bureau of Standards (NBS).

The first volume, NBS SP 260-71, "Summary of the Clinical Laboratory Standards Issued by the National Bureau of Standards" (176 pp., November 1981), describes in a tabular form 41 SRM's available in that field and includes copies of the corresponding Certificates of Analysis for further information.

The second volume, NBS SP 260-97, "Summary of the Coal, Ore, Mineral, Rock, and Refractory Standards Issued by the National Bureau of Standards" (134 pp., September 1985), describes in the same manner 39 SRM's available in that field.

The third volume, NBS SP 260-104, "Summary of the Biological and Botanical Standards Issued by the National Bureau of Standards" (68 pp., October 1985), presents in a similar manner the essential data concerning 9 SRM's issued in that field.

The present volume, NBS SP 260-105, "Summary of the Environmental Research, Analysis, and Control Standards Issued by the National Bureau of Standards" (97 pp., March 1986), describes in a tabular form the characteristic properties of 22 SRM's issued in the field of environmental analytical instrumentation and methodology. Copies of the Certificates of Analysis are reproduced in this work also, for further information.

Tables 2 and 3 contain the essential information concerning the material composition, the certification parameters, and use. Under "Remarks," additional data such as storage conditions and stability is provided. All the data and information contained in these tables were extracted from the Certificates or Certificates of Analysis issued for the SRM's included in the table. An examination of these tables gives the reader a general view of these SRM's. For more detailed information, the individual Certificates reproduced in appendix II should be consulted as well as the references cited in each Certificate. The SRM's listed in the tables include all of the environmental standards that were issued or were in preparation by the end of 1984. These SRM's are the result of the concerted efforts of a number of scientists from the NBS National Measurement Laboratory. Each Certificate lists the individuals who contributed to development of the SRM.

Appendix III provides a guide to the reader to assist in requesting NBS to develop new SRM's. A final appendix, appendix IV, is a guide to ordering SRM's.

In addition to the SRM's and their Certificates, NBS issues a series of Special Publications, called the "260 Series," that relate directly to Standard Reference Materials as stated in the preface. The list of available publications in the "260 Series" is given at the beginning of this publication.

NOTE: The use of proprietary designations in table 2 is for information only, and should not be construed as an endorsement of the product by either the Department of Commerce or the National Bureau of Standards.

For complete bibliographic reference and ordering information, see "Other NBS Publications in This Series," pp. iv.

TABLE 2. SUMMARY OF THE ENVIRONMENTAL RESEARCH, ANALYSIS, AND CONTROL STANDARDS.

SRM	Material	COMPOSITION
1579 Powdered Lead Based Paint	Collected by the Philadelphia Dept. Public Health, and sieved to a powder passing through a 325 mesh sieve.	Pb: 11.87 ± 0.04 % by wt., based on samples >0.1 g of as-received material.
1580 Organics in Shale Oil	The shale oil came from the Laramie Energy Technology Center, Laramie, Wyoming, and was collected from the Mahogany Zone of the Colorado Green River Formation.	Fluoranthene: 54; pyrene: 104; benzo[a]pyrene: 21; benzo[e]pyrene: 18; perylene: 3.4; phenol: 407; o-cresol: 385; 2,6-dimethylphenol: 175; benzol[f]quinoline: 16, µg/g.
1620a Sulfur in Residual Fuel Oil	The material is a commercial "No. 5 Heavy" residual fuel oil as defined by the American Society for Testing and Materials.	S: 4.504 <u>+</u> 0.010 wt. %.
1621b Sulfur in Residual Fuel Oil	The material is a commercial "No. 6" residual fuel oil as defined by the American Society for Testing and Materials.	S: 0.950 <u>+</u> 0.005 wt. %.
1622b Sulfur in Residual Fuel Oil	Same as for SRM 1621b.	S: 1.982 <u>+</u> 0.018 wt. %.
1623a Sulfur in Residual Fuel Oil	Same as for SRM 1620a.	S: 0.240 <u>+</u> 0.003 wt. %.
1624a Sulfur in Distillate (Diesel) Fuel Oil	The material is a commercial "No. 2-D" distillate fuel oil as defined by the American Society for Testing and Materials.	S: 0.141 <u>+</u> 0.002 wt. %.
1630 Trace Mercury in Coal	Commercial coal crushed to 210-500 micrometer particle size.	Hg: $0.13 \mu g/g \pm 1$ in the last significant figure.

By x-ray fluorescence, atomic absorption spectrometry, polarography.

In the calibration of apparatus and methods for determining Pb in paint removed from the interior surfaces of old housing.

By gas chromatography, gas chromatography/mass spectrometry, and high performance liquid chromatography. For evaluating the reliability of analytical methods used for trace organic compounds in oil materials.

Seven additional organic compounds are included for information only and are not certified.

By gravimetry, ion chromatography, and x-ray fluor-escence.

As analytical standard in the determination of total S in fuel oils and similar materials.

Four additional physical properties are indicated but are not certified; similarly, 16 trace elements are mentioned semi-quantitatively only.

As for SRM 1620a. Is valid for 3 years from date of purchase.

As for SRM 1620a.

As for SRM 1621b.

As for SRM 1621b.

As for SRM 1620a.

As for SRM 1620a.

By gravimetry and ion chromatography. Is valid for 3 years from date of purchase.

As for SRM 1620a.

By neutron activation and flameless atomic absorption spectrometry (0.14  $\mu g/g$ ). The neutron activiation procedure is described in the certificate.

As analytical standard for the determination of trace mercury in coal.

Selenium is also given for information, but is not certified (2.1  $\mu g/g$ ).

1632b Trace Elements in Coal (Bituminous)

Obtained from an underground mine that recovers coal from the Pittsburgh seam, crushed and sieved through a -60 mesh at the Humphrey No. 7 mine and coal preparation plant of the Consolidation Coal Co., Christopher Coal Co. Div., Osage, W. Va.

Material should be vacuum dried at ambient temperature for 24 hours prior to use. Values based on a minimum sample size of 250 mg. C (total) 78.11; H 5.07; N 1.56; S 1.89; Volatile matter 35.4; Al 0.855; Ca .204; Fe .759; Mg .0383; K .0748; Na .0515; Ti .0454; values % by wt. As 3.72; Ba 67.5; Cd 0.0573; Co 2.29; Cu 6.28; Pb 3.67; Mn 12.4; Ni 6.10; Rb 5.05; Se 1.29; Th 1.342; U 0.436; Zn 11.89; values μg/g. Not certified: 17 additional constituents (see certificate).

1633a Trace Elements in Coal Fly Ash Obtained from a coal fired power plant using Pennsylvania and West Virginia Coal. The ash was sieved through a No. 170 sieve.

Determined on 250 mg or more sample dried to constant weight. Al 14.3; Ca 1.11; Fe 9.40; K 1.88; Mg 0.455; Na 0.17; Si 22.8; wt. %. Sb 6.8; As 145; Cd 1.0; Cr 196; Cu 118; Hg 0.16; Ni 127; Pb 72.4; Rb 131; Se 10.3; Sr 830; Th 24.7; Tl 5.7; U 10.2; V 2.97; Zn 220;  $\mu g/g$ . Additional 15 elements determined but not certified.

1634a Trace Elements in Fuel Oil Commerical No. 6 residual fuel oil as defined by ASTM.

Determined on at least 1 g sample. Pb 2.80; Mn 0.19; Ni 29; Se 0.15; Na 87; V 56; Zn 2.7;  $\mu g/g$ . S 2.85 wt. %. Values for additional 11 elements and 4 physical properties are given but not certified.

1635 Trace Elements in Coal (Subbituminous) Subbituminous coal from Eagle Mine of the Imperial Coal Co. of Erie, Colorado; sieved through a No. 65 sieve.

Determined on at least 250 mg of dried sample. As 0.42; Cd 0.03; Cr 2.5; Cu 3.6; Pb 1.9; Mn 21.4; Ni 1.74; Se 0.9; Th 0.62; U 0.24; V 5.2; Zn 4.7; µg/g; and Fe 0.239; S 0.33 wt %. Additional 10 elements determined but not certified.

1636a, 1637a, 1638a Lead in Reference Fuel Supplied by Phillips Petroleum Co., Bartlesville, Okla. Lead was added as tetraethyl lead motor mix.

Determined on at least 1 g sample. Vial I-11.2; Vial II-18.8; Vial III-25.1; Vial IV-764 µg/g.

Analyses performed in the NBS Center for Analytical Chemistry. Estimated uncertainty 0.006 depending on constituent. For the calibration of apparatus and the evaluation of techniques employed in the analysis of coal or similar materials.

Should be kept in its original bottle. Should not be exposed to intense sources of radiation, including ultraviolet lamps or sunlight.

Using two to four different analytical procedures for each element as shown in the Certificate, on the dried sample. For calibration of apparatus and methods used in the analysis of coal ash and similar materials.

The materials should be dried as indicated in the Certificate. Stability (>3 years) not yet established.

Using two to three independent analytical procedures for each element.

For calibration of apparatus and methods used in the analysis of fuel oils and similar materials for trace elements.

Store in tightly sealed bottle. Certificate valid for 3 years from date of purchase.

Using two to three independent analytical procedures for each element. For sample drying, see the Certificate. For calibration of apparatus and techniques used in trace element analysis of coal and similar materials. Store in tightly sealed bottle in a cool, dark place. Long term (>1 year) stability not yet established.

By isotope dilution mass spectrometry. SRM 1636a is made from Vials I, II, III, and IV. SRM 1637a is made from Vials I, II, and III. SRM 1638a is made from Vial IV.

For calibration of instruments and techniques used for the analysis of Pb in gasoline. The vials should be stored at 10-30 °C in darkness, and not reused after first opening.

SRM	Material	Composition
1641b Mercury in Water - μg/mL	This SRM was prepared at NBS and is delivered in 6 ampoules of 20 mL each.	Hg: 1.52 <u>+</u> 0.04 μ/mL.
1642b Mercury in Water - ng/mL	Same as SRM 1641b.	Hg: 1.49 <u>+</u> 0.06 ng/mL.
1643b Trace Elements in Water	Prepared at the U.S. Geological Survey, National Water Quality Laboratory, Arvada, Colo., by using high-purity reagents and sterilization.	Ba 44; Be 19; Cd 20; Cr 18.6; Co 26; Cu 21.9; Fe 99; Pb 23.7; Mn 28; Mo 85; Ni 49; Se 9.7; Ag 9.8; Sr 227; Tl 8; V 45.2; Zn 66 ng/g. Additional 3 elements determined but not certified.
1644 Generator Columns for Polynuclear Aromatic Hydrocarbons	Three 50 cm x 0.6 cm coiled stainless steel tubes each packed with fine quintus quartz coated with 0.5 wt. % of polynuclear aromatic hydrocarbon (PAH).	At 20 °C: anthracene 30.7; benzo(a)anthracene 6.45; benzo(a)pyrene 1.13, all in µg/Kg.
1645 River Sediment	Deposit dredged from the bottom of Indiana Harbor Canal, Gary, Ind., freeze-dried, sieved (No. 80 screen) and radiation-sterilized.	A1 2.26; Cr 2.96; Fe 11.3; K 1.26; Mg 0.74; Na 0.54; Zn 0.172, wt %. Cd 10.2; Cu 109; Co 10.1; Pb 714; Mn 785; Hg 1.1; Ni 45.8; Tl 1.44; Th 1.62; U 1.11; V 23.5; µg/g. Additional 13 values deter- mined but not certified.
1646 Estuarine Sediment	Dredged from the Chesapeake Bay, freeze-dried, radiation- sterilized and sieved (No. 100 sieve).	A1 6.25; Ca 0.83; Fe 3.35; Mg 1.09; P 0.054, wt. %. As 11.6; Cd 0.36; Cr 76; Co 10.5; Cu 18; Pb 28.2; Mn 375; Hg 0.063; Ni 32; V 94; Zn 138, µg/g.
1648 Urban Particulate Matter	Urban particulate matter was collected in St. Louis, Mo., over a period of 12 months.	Al 3.42; Fe 3.91; K 1.05; Pb 0.655; Na 0.425; Zn 0.476, wt. %. As 115; Cd 75; Cr 403; Cu 609; Ni 82; Se 27; U 5.5; V 140 µg/g. Non-certified values are given for 26 elements.

By atomic absorption spectrometry and neutron activation. Use of blank samples is necessary.

For primary calibration of instruments and techniques and as a spike sample in method-of-addition procedures for the determination of Hg in natural waters.

Stability limited to 1 year from date of purchase.

Same as SRM 1641b.

Same as SRM 1641b.

Should be used without dilution. For precautions in use see Certificate.

At least two from the nine analytical procedures employed were used for the determination of each element.

For evaluating the accuracy of trace element determination in fresh water and for instrument calibration. The certification is valid for two years from the shipping date. For precautions in use see Certificate.

Performed by two independent analytical procedures. Concentrations for the three PAH at other temperatures (10-  $^{30}$  °C) are given in the Certificate.

This SRM is intended to provide accurate concentrations of the three PAH.

For further properties, use, and stability, see Certificate.

Six indepedent analytical procedures and 100 mg to 1 g of sample were used for certification.

For calibration of apparatus and methods used in the analysis of river sediments or similar materials.

For details on stability, use, and homogeneity, see Certificate. Data valid for 5 years from date of purchase.

By 6 indepedent analytical procedures on 500 mg or more dried sample, using two to four different procedures for each element.

For calibration of instrumentation and evaluatin of analytical methods in sediments and similar matrices. Certified data valid for 5 years after date of shipping.

On 100 mg or more of dried sample using 9 independent analytical procedures.

Calibration of apparatus and evaluation of methods used in the analysis of atmospheric particulate matter and similar matrices.

Certification valid for 5 years from date of purchase, for samples kept in the original bottle at  $10-30\,^{\circ}\text{C}$  in a desiccator and in dark.

#### Analyzed Liquids and Solids

#### MULTI-ELEMENT

#### Concentrations:

Values expressed as microgram per gram, except:

Weight Percent -- %

Nanogram per gram -- italics

Parenthesis indicates elements not certified and given for information only.

SRM	Туре	Unit Size	Al	Sb	As	Ва
1632b	Trace Elements in Coal					
	(Bituminous)	75 g	0.855%	(0.24)	3.72	67.5
1633a	Trace Elements in Coal			, , ,		
	Fly Ash	75 g	14.3%	6.8	145	(0.15%)
1634a	Trace Elements in Fuel Oil	100 mL			(0.12)	
1635	Trace Elements in Coal					
	(Subbituminous)	75 g	(0.32%)	(0.14)	0.42	
1643Ъ	Trace Elements in Water					
	(ng/g)	950 mL			(49)	44
1645	River Sediment	70 g	2.26%	(51)	(66)	
1646	Estuarine Sediment	75 g	6.25%	(0.4)	11.6	
1648	Urban Particulate	2 g	3.42%	(45)	115	(737)

SRM	Ве	Bi	В	Br	Cd	Ca	С	Ce
1632b				(0.17)	0.0573	0.204%	78.11%	(9)
1633a	(12)				1.00	1.11%		(180)
1634a	(0.006)			(<1)	(0.002)	(16)		
1635					0.03			(3.6
1643b	19	(11)	(94)		20			
1645					10.2	(2.9%)		
1646	(1.5)				0.36	0.83%		(80)
1648				(500)	75			(55)

SRM	Cs	C1	Cr	Со	Cu	Eu	F
1632ъ	(0.44)	(1260)	(11)	2.29	6.28	(0.17)	
L633a	(11)		196	(46)	118	(4)	
L634a		(31)	(0.7)	(0.3)			
.635			2.5	(0.65)	3.6	(0.06)	
.643b			18.6	26	21.9		
645			2.96%	10.1	109		(0.09%)
646	(3.7)		76	10.5	18	(1.5)	
.648	(3)	(0.45%)	403	(18)	609	(0.8)	

SRM	Ga	Ge	Hf	Н	In	I	Fe	La
- ( 0 0 1			(0, (0)	5 0 7 %			0.750%	<b>(5.1)</b>
1632Ъ			(0.43)	5.07%			0.759%	(5.1)
1633a	(58)		(8)				9.4%	
1634a							(31)	
1635	(1.05)		(0.29)				0.239%	
1643Ъ							99	
1645							11.3%	(9)
1646		(1.4)					3.35%	
1648			(4.4)		(1.0)	(20)	3.91%	(42)

Table 3. Continued.

SRM	Pb	Li	Mg	Mn	Hg	Мо	Ni	N
1632Ъ	3.67	(10)	0.0383%	12.4		(0.9)	6.10	1.56
1633a	72.4		0.455%	179	0.16	(29)	127	
1634a	2.80			0.19	(<0.002)	(0.12)	29	
1635	1.9			21.4			1.74	
1643Ъ	23.7			28		85	49	
1645	714		0.74%	785	1.1		45.8	
1646	28.2	(49)	1.09%	375	0.063	(2.0)	32	
1648	0.655%		(0.8%)	(860)			82	

SRM	P	K	Rb	Sm	Sc	Se	Si	Ag
1632ь		0.0748%	5.05	(0.87)	(1.9)	1.29	(1.4%)	
1633a		1.88%	131		(40)	10.3	22.8%	
1634a						0.15		
1635					(0.63)	0.9		
1643Ъ						9.7		
1645		1.26%			(2)	(1.5)		
1646	0.054%	(1.4%)	(87)		(10.8)	(0.6)	(31%)	
1648		1.05%	(52)	(4.4)	(7)	27		(6)

SRM	Na	Sr	S	Te	Tl	Th	Ti	W
1632b	0.0515%	(102)	1.89%			1.342	0.0454%	(0.48)
1633a	0.17%	830	(0.18%)		5.7	24.7	(0.8%)	
1634a	87		2.85%					
1635	(0.24%)		0.33%			0.62	(0.02%)	
1643Ъ		227			8.0			
1645	0.54%		(1.1%)		1.44	1.62		
1646	(2.0%)		(0.96%)	(0.5)	(0.5)	(10)	(0.51%)	
1648	0.425%		(5.0%)			(7.4)	(0.40%)	(4.8)

SRM	U	V	Zn
1632b	0.436	(14)	11.89
1633a	10.2	297	220
1634a		56	2.7
1635	0.24	5.2	4.7
1643b		45.2	66
1645	1.11	23.5	0.172%
1646		94	138
1648	5.5	140	0.476%

Table 5. Continued.

# Analyzed Liquids and Solids

#### SINGLE ELEMENT

SRM	Туре	Unit Size	Lead	Sulfur	Mercury
1579	Powdered Lead Base Paint	35 g	11.87%		
1620a	Sulfur in Residual Fuel Oil	100 mL		4.504%	
1621b	Sulfur in Residual Fuel Oil	100 mL		0.950%	
1622b	Sulfur in Residual Fuel Oil	100 mL		1.982%	
1623a	Sulfur in Residual Fuel Oil	100 mL		0.240%	
1624a	Sulfur in Residual Fuel Oil	100 mL		0.141%	
1630	Trace Mercury in Coal	50 mg			0.13 μg/g
1641b	Mercury in Water - μg/mL	120 mL			$1.52  \mu \text{g/mL}$
1642Ъ	Mercury in Water - ng/mL	950 mL			1.49 ng/mL

		*		
		Element	Nominal	
SRM	Туре	Certified	Concentration	No. Units
1636a	Lead in Reference Fuel	Pb	0.03, 0.05, 0.07, and	3 vials each
			2.0 g/gal	
1637a	Lead in Reference Fuel	Pb	0.03, 0.05, 0.07 g/gal	4 vials each
1638a	Lead in Reference Fuel	Pb	2.0 g/gal	12 vials

# Organic Constituents

SRM	Туре	Unit of Issue
1580 1644	Shale Oil Polyaromatic Hydrocarbon Generator Columns	Set of 5-2 mL ampoules Set of 3 columns

	SRM 1580	SRM 1644
Constituents	(μg/g)	(μg/kg)
Anthracene		30.7
Benzo[a]anthracene		6.45
Benzo[a]pyrene	21	1.13
Benzo[e]pyrene	18	
Fluoranthene	54	
o-Cresol	385	
Phenol	407	
Perylene	3.4	
Pyrene	104	
2,6-Dimethylphenol	175	
Benzo $[f]$ quinoline	16	

# Appendix I.

# Alphabetical ricer by Standard Telescope Material Name

Name	SRM	Name	SRM
Acetanilide	141c	Aluminum, Freezing Point Standard	44f
Acid Open-Hearth Steel, 0.2% Carbon	19G	Aluminum, Magnetic Gram	763
Acid Potassium Phthalate	84 j	Susceptibility	
AISI 1045 Steel	20g	Aluminum Oxide, Melting Point	742
AISI 4340 Steel	36l	Aluminum Rod Ultra Purity	RM 1R
AISI 4340 Steel	1261a	Aluminum-26 Radioactivity Standard	4229
AISI 94B17 Steel (Modified)	362	Americium-241 Alpha-Particle	4904F
AISI 94B17 Steel (Modified)	1262a	Standard	
Albacore Tuna	RM 50	Americium-241 Gamma-ray Standard	4213
Alkali Lead Silicate Glass	712	Ammonium Dihydrogen Phosphate	194
Alpha Quartz	1878	Angiotensin I (Human)	998
Alumina (Reduction Grade)	699	Anisic Acid	142
Alumina Silicate Glass	714	Anticonvulsant Drug Level Assay	1599
Aluminosilicate Glass	715	Standard	
Aluminum Alloy	85B	Antiepilepsy Drug Level Assay	900
Aluminum Alioy 6011 (Modified)	858	Standard	
Aluminum Alloy 6011 (Modified)	1258	Antimony-125-Tellurium-125m,	4275B
Aluminum Alloy 7075	859	Europium-154, Europium-155 Mixed-	
Aluminum Alloy 7075	1259	Radionuclide Point-Source Standard	427(D
Aluminum Block, Eddy Current	1860	Antimony-125-Tellurium-125m, Europium-154, Europium-155 Mixed-	4276B
Conductivity	10/1	Radionuclide Solution Standard	
Aluminum Block, Eddy Current	1861	A.O.H., 0.4C Spectrographic Steel	413
Conductivity Aluminum Block, Eddy Current	1862	Standard	413
Conductivity	1002	Argillaceous Limestone	1C
Aluminum Block, Eddy Current	1863	Arsenic Trioxide Reductometric	83d
Conductivity	1003	Standard	054
Aluminum Brass Standard for	1118	Assay-Isotopic Standard for Potassium	985
Optical Emission and X-ray		Assay-Isotopic Standard for Rhenium	989
Spectroscopic Analysis		Assay-Isotopic Standard for Silicon	990
Aluminum Brass Standard for	C1118	Assay-Isotopic Standard for Strontium	987
Optical Emission and X-ray		2% Austenite in Ferrite	488
Spectroscopic Analysis		5% Austenite in Ferrite	485a
Aluminum Brass Standard for	1119	15% Austenite in Ferrite	486
Optical Emission and X-ray		30% Austenite in Ferrite	487
Spectroscopic Analysis			
Aluminum Brass Standard for	C1119	·	
Optical Emission and X-ray			
Spectroscopic Analysis			
Aluminum Casting Alloy 356	855		
Aluminum Casting Alloy 380	856		
Aluminum Cube Ultra Purity	RM 1C		
Aluminum 2-Ethylhexanoate	1075a		

Name	SRM	Name	SRM
Austenitic Stainless Steel, Thermal	1460	Beryllium on Filter Media	2675
Conductivity and Electrical		Bessemer Steel (Simulated)	8j
Resistivity		0.1% Carbon	0.4.5
Austenitic Stainless Steel, Thermal	1461	Bilirubin	916 1080a
Conductivity and Electrical Resistivity		Bis(1-phenyl-1, 3-butanediono) copper (II)	1080a
Austenitic Stainless Steel, Thermal	1462	Bis(1-phenyl-1, 3-butanediono)	1052b
Conductivity and Electrical	1402	oxovanadium (IV)	10320
Resistivity		Black Porcelain Enamel for Directional	2021
Barium Crown Glass	713	Hemispherical Reflectance	
Barium Cyclohexanebutyrate	1051b	Black Porcelain Enamel for Directional	2022
Barrium-133 Radioactivity Point-Source	4241B	Hemispherical Reflectance	1142
Standard	4251D	Blast Furnace Iron Standard	1143a
Barium-133 Radioactivity Standard Basalt Rock	4251B 688	(Chill Cast White) Blast Furnace Iron Standard	1144a
Base Oil	1083	(Chill Cast White)	11 <del>44</del> a
Basic Electric Spectrographic Steel	404a	B.O.H., 0.4C Spectrographic Steel	417a
Standard		Standard	
Basic Open-Hearth Steel, 0.1% Carbon	15g	Boric Acid	951
Basic Open-Hearth Steel, 0.1% Carbon	335	Boron-Doped Silicon Slices for	1521
Basic Open-Hearth Steel, 0.1% Carbon	1228	Resistivity Measurements	
Basic Open-Hearth Steel, 0.2% Carbon	11h	Borosilicate Glass	93a
Basic Open-Hearth Steel, 0.4% Carbon	12H 152A	Borosilicate Glass	623 717
Basic Open-Hearth Steel, 0.5% Carbon Basic Open-Hearth Steel, 0.8% Carbon	132A 14f	Borosilicate Glass Borosilicate Glass	1825
Basic Open-Hearth Steel, 1% Carbon	1227	Borosilicate Glass, Thermal Expansion	731
(Disk)		Bovine Liver	1577a
Basic Open-Hearth Steel, 1.1% Carbon	16f	Bovine Serum Albumin	926
Basic Open-Hearth Steel, 1.1% Carbon	337	Bovine Serum Albumin (7% Solution)	927
0.4C Basic Oxygen Furnace Steel	178	Branched Polyethylene	1476
Bauxite (Arkansas)	69b	Brewers Yeast	1569
Bauxite (Dominican)	697 698	Bright Copper Microhardness	1894
Bauxite (Jamaican) Bauxite (Surinam)	696	Standard Bright Nickel Microhardness Standard	1895
Benzene in Nitrogen	1805	Bromobenzoic Acid	2142
Benzene in Nitrogen	1806	Burnt Refractory	76a
Benzene Permeation Device	1911	Burnt Refractory	77a
Benzoic Acid	140b	Burnt Refractory	78a
Benzoic Acid	350a	Cadmium Cyclohexanebutyrate	1053a
Benzoic Acid Calorimetric Standard	39i 373f	Cadmium, Vapor Pressure	746
Benzothiazyl Disulfide Rubber Compound	3/31	Calcium Carbonate Calcium 2-Ethylhexanoate	915 1074a
Beryllium-Copper Standard	1122	Calcium in Low-Alloy (Silicon) Steel	1254
Beryllium-Copper Standard	C1122	Calcium Molybdate	71
Beryllium-Copper Standard	C1123	Calibrated Glass Beads	1004
		Calibrated Glass Beads	1017a
		Calibrated Glass Beads	1018a
		Calibrated Glass Spheres	1003a
		Carbon Dioxide in Air Carbon Dioxide in Air	1670 1671
		Carbon Dioxide in Air	1672
		Carbon Dioxide in Nitrogen	1674b
		Carbon Dioxide in Nitrogen	1675b
		Carbon Dioxide in Nitrogen	2619a
		(Combustion Efficiency Gas Standard)	
		Carbon Dioxide in Nitrogen	2620a
		(Combustion Efficiency Gas Standard)	

Name	SRM	Name	SRM
Carbon Dioxide in Nitrogen	2621a	Catalyst Package for Lubricant	1817
(Combustion Efficiency Gas Standard) Carbon Dioxide in Nitrogen	2622a	Oxidation Centerline Drawings for Optical	1901
(Combustion Efficiency Gas Standard)		Character Recognition, B	
Carbon Dioxide in Nitrogen	2623a	Characters	***
(Combustion Efficiency Gas Standard)	2624a	Centroid Color Chart	2106 2107
Carbon Dioxide in Nitrogen (Combustion Efficiency Gas Standard)	2024a	Centroid Color Kit Cesium-137, Barium-137m Point-Source	4200B
Carbon Dioxide in Nitrogen	2625a	Radioactivity Standard	12001
(Combustion Efficiency Gas Standard)		Cesium-137, Barium-137m Point-Source	4207
Carbon Dioxide in Nitrogen	2626a	Radioactivity Standard	
(Combustion Efficiency Gas Standard)	2632	Cesium-134 Badioactivity Standard	4233B 4250B
Carbon Dioxide in Nitrogen (Mobile Source Emission Gas Standard)	2032	Cesium-134 Radioactivity Standard Channel Black Rubber Compound	375g
Carbon Dioxide in Nitrogen (Mobile	2633	Chlorine-36 Beta-ray Standard	4943
Source Emission Gas Standard)		Chlorine-36 Radioactivity Standard	4422L
Carbon Monoxide in Air (Ambient	2612a	Chlorobenzoic Acid	2144
Air Quality Gas Standard)	2613a	Chrome Refractory	103a
Carbon Monoxide in Air (Ambient Air Quality Gas Standard)	2013a	Chromium-Molybdenum-Aluminum Steel	106B
Carbon Monoxide in Air (Ambient	2614a	Chromium-Molybdenum Steel	36b
Air Quality Gas Standard)		Chromium-Molybdenum Steel	133B
Carbon Monoxide in Nitrogen	1677c	Chromium-Nickel-Molybdenum Steel	139b
Carbon Monoxide in Nitrogen	1678c 1679c	Chromium-Nickel-Molybdenum Steel	1222 339
Carbon Monoxide in Nitrogen Carbon Monoxide in Nitrogen	1680b	17Chromium-9 Nickel-0.2 Selenium Steel Chromium-Nickel Spectrographic Steel	408a
Carbon Monoxide in Nitrogen	1681b	Standard	1004
Carbon Monoxide in Nitrogen (Mobile	2635	15Chromium-7 Nickel Steel	344
Source Emission Gas Standard)		16 Chromium-4 Nickel Steel	345
Carbon Monoxide in Nitrogen (Mobile	2636	Chromium-51 Radioactivity Standard	4400L-F 163
Source Emission Gas Standard) Carbon Monoxide in Nitrogen (Mobile	2637	Chromium Steel Chromium-Tungsten Steel	155
Source Emission Gas Standard)	2037	Chromium-Vanadium Spectrographic	407a
Carbon Monoxide in Nitrogen (Mobile	2638	Steel Standard	
Source Emission Gas Standard)		Cholesterol	911a
Carbon Monoxide in Nitrogen (Mobile	2639	Chrysotile Asbestos Fibers	1876 1572
Source Emission Gas Standard) Carbon Monoxide in Nitrogen (Mobile	2640	Citrus Leaves Clinical Laboratory Thermometer	934
Source Emission Gas Standard)	2640	Cobalt Cyclohexanebutyrate	1055ь
Carbon Monoxide in Nitrogen (Mobile	2641	Cobalt-Molybdenum-Tungsten Steel	153A
Source Emission Gas Standard)		Cobalt-57 Radioactivity Standard	4408L-C
Carbon Monoxide in Nitrogen (Mobile	2642	Cobalt-60 Radioactivity Standard Commercial Bronze Standard for	4915D 1115
Source Emission Gas Standard) Carbon-14 Radioactivity Standard	1245	Optical Emission and X-ray	1113
Carbon-14 Radioactivity Standard  Carbon-14 Radioactivity Standard	4245 4246	Spectroscopic Analysis	
Carbon Steel	1224	Commercial Bronze Standard for	C1115
Carbon Steel, 0.6%	13g	Optical Emission and X-ray	
Cast Iron	4k	Spectroscopic Analysis Commercial Bronze Standard for	1117
Cast Iron Cast Iron	5L	Optical Emission and X-ray	1116
Cast Iron	6g 7G	Spectroscopic Analysis	
Cast Iron Car Wheel	122h		
Cast Steel 3	C1173		
Cast Steel Standard	1138a		
Cast Steel Standard	1139a		

Name	SRM	Name	SRM
Commercial Bronze Standard for Optical Emission and X-ray	C1116	Cupro-Nickel, 10% (CDA 706) High Purity	874
Spectroscopic Analysis		Cystine	143c
Commercial Bronze Standard for	1117	Dextrose	41b
Optical Emission and X-ray		D-Glucose	917
Spectroscopic Analysis		Dibutyltin Bis(2-ethylhexanoate)	1057b
Commercial Bronze Standard for Optical Emission and X-ray Spectroscopic Analysis	C1117	Didymium Glass Filter for Checking the Wavelength Scale of Spectrophotometers	2009
Common Lead Isotopic Standard	981	Didymium Glass Fitler for Checking	2010
Copper Concentrate	332	the Wavelength Scale of	2010
Copper Heat Capacity Test Specimen	RM5	Spectrophotometers	
Copper-Nickel-Chromium Cast Iron	115A	Disodium Hydrogen Phosphate	186IIc
Copper Ore, Mill Heads	330	Disodium Hydrogen Phosphate	2186II
Copper Ore, Mill Tails	331	D-Mannitol	920
Copper-Thermal Expansion	736a	Dolomitic Limestone	88a
Copper, Secondary Freezing Point	45d	Doped Platinum	681L1
Standard	73u	Doped Platinum	681L2
Cortisol (Hydrocortisone)	921	Ductile Cast Iron	341
Creatinine	914	Electrical Residual Resistivity Ratio	769
Cr-Mo Low Alloy Steel	1270	Standard Residual Resistivity Ratio	707
Cr-Mo Steel (ASTM A-213)	291	Electrolytic Iron	365
Cr-Mo (SAE 4140) Spectrographic	414	Electrolytic Iron	1265a
Steel Standard	714	Electrolytic Iron, Thermal	1463
Cr-Mo (SAE 4150) Spectrographic	427	Conductivity and Electrical	1405
Steel Standard	721	Resistivity	
Cr-Mo (SAE X4130) Spectrographic	418a	Electrolytic Iron, Thermal	1464
Steel Standard	7104	Conductivity and Electrical	1404
Cr-Ni-Mo Steel (AISI 8620)	293	Resistivity	
18Cr-10Ni Steel (AISI 304L)	101f	Electronic and Magnetic Alloy	1159
Cr-V Steel (Modified)	363	Standard	1137
Cr-V Steel (Modified)	1263a	Electronic and Magnetic Alloy	1160
Cr-V Steel (SAE 6150)	30f	Standard	1100
Crystalline Potassium Dichromate	935	Enriched Boric Acid	952
Crystalline Potassium Iodide,	2032	Equal-Atom Lead Isotopic Standard	982
Heterochromatic Stray Radiant	2032	Estuarine Sediment	1646
Energy Standard		Europium-152 Point-Source Standard	4218E
Crystalline (Ruby) Electron	2601	Europium-152 Radioactivity Standard	4370B
Paramagnetic Resonance	2001	Extra Dense Lead Glass	709
Absorption Intensity Standard		Fe-Cr-Ni Alloy Microprobe Standard	479a
Cupro-Nickel (CDA 706)	1275	Fe-3Si Alloy Microprobe Standard	483
Cupro-Nickel (CDA 715)	1276	Feldspar	70a
Cupro-Nickel, 10% (CDA 706) Doped	875	Feldspar	99a
	0.0	Ferrochromium (Low Carbon)	196
		Ferrochromium Silicon	689
		Ferroniobium	340
		Ferrophosphorus	90
		Ferrosilicon	58a
		Ferrosilicon	59a
		Ferrosilicon (75% Si)	195
		First Surface Aluminum Mirror for	2003a
		Specular Reflectance	
		First Surface Mirror, Gold on Glass	2008a

Name	SRM	Name	SRM
Fission Track Glass Standard	961	Gold-198 Radioactivity Standard	4405L-B
Fission Track Glass Standard	962a	Gold-Silver Wires for Microprobe	481
Fission Track Glass Standard	963a	Analysis	
Fission Track Glass Standard	964	Gold, Vapor Pressure	745
Flint Clay	97a	Gray Cast Iron	334
Fluorobenzoic Acid	2143	Halocarbons (in methanol) for Water	1639
Fluorspar	79a	Analysis	
Free-Cutting Brass	1103	High-Alloy Steel (A-743)	C1288
Free-Cutting Brass	C1104	High-Alloy Steel (AISI 310 Mod.)	C1287
Freeze-Dried Urine	2670	High-Alloy Steel, (AISI 414 Mod.)	C1289
Freeze-Dried Urine Certified	2671a	High-Alloy White Cast	892
for Fluoride		High-Alloy White Cast Iron	890
Freeze-Dried Urine Certified	2672a	High-Alloy White Cast Iron	891
for Mercury		High-Carbon Ferrochromium	64c
Fused-Silica Thermal Expansion	739	High-Carbon Ferromanganese	68c
Gadolinium-148 Alpha-Particle	4907	High-Carbon Steel (Modified)	364
Standard		High-Carbon Steel (Modified)	1264a
Gallium Melting-Point Standard	1968	High-Grade Fluorspar	180
Gallium-67 Radioactivity Standard	4416L-D	High-Nickel Steel	126c
Gas Furnace Black Rubber Compound	382a	High-Nickel Steel	1158
Gasometric Set (1095-1099)	1089	High-Purity Gold	685
Gasometric Standard for Unalloyed	357	High-Purity Platinum	680L1A
Zirconium		High-Purity Platinum	680L2A
Gasometric Standard for Unalloyed	358	High-Purity Platinum Thermoelement	1967
Zirconium		High-Purity Zinc	682
Generator Columns for Polynuclear	1644	High-Silicon Steel	179
Aromatic Hydrocarbons		High-Silicon Steel	1134
Gilding Metal	1112	High-Silicon Steel	1135
Gilding Metal	C1112	High-Silicon Steel (Calcium Bearing)	125b
Gilding Metal	1113	High-Sulfur Steel	105
Gilding Metal	C1113	High-Sulfur Steel	129c
Gilding Metal	1114	High-Sulfur Steel	1136
Gilding Metal	C1114	High Temperature Alloy A286	348
Glasses for Microchemical Analysis	1871	High Temperature Alloy M308	1197
Glasses for Microchemical Analysis	1872	High Temperature Alloy L605 and	S1199
Glasses for Microchemical Analysis	1873	S816	120/ 2
Glasses for Microchemical Analysis	1874	High-Temperature Alloy	1206-2
Glasses for Microchemical Analysis	1875	High-Temperature Alloy	1207-1
Glass Fibers for Microanalysis	RM 31	High-Temperature Alloy	1207-2
Glass Filter for Transmittance	2030	High-Temperature Alloy	1208-1
Measurement	0205	High-Temperature Alloy	1208-2
Glass Filters for Spectrophotometry	930D	Homogeneous River Sediment for Radioactivity Measurements	RM 45B
Glass Fluorescence Source	477	Human Liver, Environmental	4352
Glass Sand	81a	Radioactivity	4332
Glass Sand	165a	Human Lung, Environmental	4351
Glass Spheres Gold Coating on Glass Sealing Alloy	1019a 1398a	Radioactivity	7331
Gold Coating on Nickel	1379	Human Serum	909
Gold Coating on Nickel	1380		, ,
Gold Coating on Nickel	1399b		
Gold-Copper Wires for Microprobe	482		
Analysis	102		
Gold-195 Radioactivity Standard	4421L		

Name	SRM	Name	SRM
Hydrogen in Unalloyed Titanium	352b	Iron Ore (Sibley)	27f
Hydrogen in Unalloyed Titanium	1086	Iron Ore Concentrate (Canada)	690
Hydrogen in Unalloyed Titanium	1087	Iron-59 Radioactivity Standard	4411L-B
Hydrogen in Unalloyed Titanium	1088	Isobutylene-Isoprene (Butyl) Rubber	1495
Hydrogen-3 Radioactivity Standard	4361	Isobutylene-Isoprene (Butyl) Rubber	388L
Hydrogen-3 Radioactivity Standard	4926C	Isotopic Standard for Bromine	977
Hydrogen-3 Toluene Radioactivity	4947	Isotopic Standard for Chlorine	975
Standard		Isotopic Standard for Chromium	979
4-Hydroxy-3 methoxy-DL-mandelic	925	Isotopic Standard for Copper	976
Acid (VMA)		Isotopic Standard for Magnesium	980
ICTA High Temperature Set	GM 760	Isotopic Standard for Silver	978
Differential Thermal Analysis		Krypton-85 Gaseous Radioactivity	4308C
ICTA Low Temperature Set Differen-	GM 757	Standard	
tial Thermal Analysis		Krypton-85 Radioactivity Standard	4235
ICTA Mod Temperature Set Differen-	GM 759	Krypton-85 Radioactivity Standard	4935C
tial Thermal Analysis		Lead-Barium Glass	89
ICTA Mid Temperature Set Differen-	GM 758	Lead-Base Bearing Metal	53e
tial Thermal Analysis		Lead-Base Bearing Metal	1132
ICTA Polystyrene Differential	GM 754	Lead Cyclohexanebutyrate	1059c
Thermal Analysis		Lead in Reference Fuel	1636a
ICTA Thermogravimetry Set	GM 761	Lead in Reference Fuel	1637a
Incoloy, 901 and Hastelloy X	S1198	Lead in Reference Fuel	1638a
Inconels, Alloy 600 (Chips)	864	Lead Nitrate	928
Inconels, Alloy 600 (Solid)	1244	Lead on Filter Media	2674
Inconels, Alloy 625 (Chips)	865	Lead-203 Radioactivity Standard	4420L
Inconels, Alloy 625 (Solid)	1245	Lead, Secondary Freezing Point	49e
Incoloy, Alloy 800 (Chips)	866	Standard	100=
Incoloy, Alloy 800 (Solid)	1246	Lead-Silica Glass	1827
Incoloy, Alloy 825 (Chips)	867	Lead-Silica Glass (Viscosity)	711
Incoloy, Alloy 825 (Solid)	1247	Lead-Silica Glass for dc Volume	624
Indium-111 Radioactivity Standard	4417L-C	Resistivity	774
Ingot Iron Spectrographic Steel Standard	420a	Lead-Silica Glass for Dielectric Constant	
Intermediate Purity Selenium	726	Lead 206 Spike Assay and Isotopic	991
Intermediate-Purity Zinc	728	Solution Standard	1035
Iodine-123 Radioactivity Standard	4414L-C	Leaded-Tin Bronze Alloy	1035
Iodine-125 Radiactivity Standard	4407L-H	Light-Sensitive Paper	700d
Iodine-129 Radioactivity Standard	4949B	Light-Sensitive Paper	701d
Iodine-131 Radioactivity Standard	4401L-I	Light-Sensitive Plastic Chip	703
Iron Foil Mössbauer Standard	1541	Linear Polyethylene	1475
Iron-55 Low-Energy Photon Standard	4260C	Linear Polyethylene	1482
Iron Metal (Clinical Standard)	937	Linear Polyethylene	1483
Iron Ore (Labrador)	692	Linear Polyethylene	1484
Iron Ore (Nimba)	693	Linerboard, Standard for Tape Adhesion Testing	1810
		Liquid Absorbance Standard for Ultraviolet and Visible	931c
		Spectrophotometry	004
		Lithium Carbonate	924
		Lithium Ore	181
		Lithium Ore	182
		Lithium Ore	183
		Low-Alloy Steel, (AISI 4130)	1225
		Low Alloy Steel	1226
		Low Alloy Steel (A242 Mod.)	C1285
		Low-Alloy Steel, AISI 4130	72g 1269
		Low Alloy Steel (AISI 1526, Modified) Low-Alloy Steel (Hy 80)	1286

Name	SRM	Name	SRM
Low-Alloy Steel Set (661-665)	S668	Naval Brass Standards for Optical	1108
Low-Carbon Silicon Steel	131c	Emission and Spectroscopic	1100
Low-Carbon Silicon Steel	1036	Analysis	
Low-Carbon Stainless Steel (AISI	166c	Naval Brass Standards for Optical	C1108
316L)	1000	Emission and Spectroscopic	C1100
Magnesium-base Alloy	171	Analysis	
Magnesium Cyclohexanebutyrate	1061c	Neutral Glass	716
Magnesium Gluconate Dihydrate	929	Neutron Density Monitor Wire	953
Magnetic Coating on Magnetic	1365a	Nickel-Chromium Cast Iron	82b
Substrate (Nickel on Steel)	1505a	Nickel-Chromium-Molybdenum Cast Iron	
Magnetic Coating on Magnetic	1366a	Nickel-Chromium Steel	32E
Substrate (Nickel on Steel)	1300a	Nickel-Copper Alloy	882
Magnetic Coating on Non-Magnetic	1367a	Nickel Cyclohexanebutyrate	1065b
Substrate (Nickel and Chromium	1307a	Nickel Oxide, No. 1	671
on brass		Nickel Oxide, No. 2	672
Magnetic Tape, High Density	6250	Nickel Oxide, No. 3	673
Manganese Fluoride, Magnetic Gram	766	Nickel-63 Radioactivity Standard	4226
Susceptibility	700	Nickel Silver (CDA 762)	879
Manganese Ore	25d	Nickel Siver (CDA 770)	880
Manganese-54 Point-Source	4997E	Nickel Spectrographic Steel Standard	409b
Radioactivity Standard	17772	Nickel Sphere, Magnetic Moment	772
Manganese-54 Radioactivity Standard	4257	Nickel Steel	33d
Manganese Steel	100B	Ni-Cr-Mo-V Steel	1173
Manganous Cyclohexanebutyrate	1062b	Nicotinic Acid	148
Maraging Steel	1156	Niobium-94 Gamma-ray Standard	4201B
Metal on Quartz Filters for	2031	Nitric Oxide in Nitrogen	1683b
Spectrophotometry	2001	Nitric Oxide in Nitrogen	1684b
Metals on Filter Media	2676b	Nitric Oxide in Nitrogen	1685b
Methane in Air	1658a	Nitric Oxide in Nitrogen	1686b
Methane in Air	1659a	Nitric Oxide in Nitrogen	1687b
Methane in Air	1660a	Nitric Oxide in Nitrogen (Mobile	2627
Medium Manganese Spectrographic	405a	Source Emission Gas Standard)	2027
Steel Standard		Nitric Oxide in Nitrogen (Mobile	2628
Mercaptobenzothiazole	383a	Source Emission Gas Standard)	
Mercury, Freezing Point	743	Nitric Oxide in Nitrogen (Mobile	2629
Mercury-203 Radioactivity Standard	4418L	Source Emission Gas Standard)	
Mercury in Water, μg/mL	164lb	Nitric Oxide in Nitrogen (Mobile	2630
Mercury in Water, ng/mL	1642b	Source Emission Gas Standard)	
Microcopy Resolution Test Chart	1010a	Nitric Oxide in Nitrogen (Mobile	2631
Microprobe Standard - Cartridge Brass	478	Source Emission Gas Standard)	
Mineral Glasses for Microanalysis	470	Nitrogen Dioxide in Air (Stationary	2653
Molybdenum Concentrate	333	Source Emission Gas Standard)	
Molybdenum, Heat Capacity	781	Nitrogen Dioxide in Air (Stationary	2654
Molybdenum-99 Radioactivity	4412L-H	Source Emission Gas Standard)	
Standard		Nitrogen Dioxide in Air (Stationary	2655
Molybdenum-Tungsten-Chromium-	134A	Source Emission Gas Standard)	
Vanadium Steel		Nitrogen Dioxide in Air (Stationary	2656
Naval Brass Standards for Optical	1106	Source Emission Gas Standard)	
Emission and Spectroscopic		Nitrogen Dioxide Permeation Device	1629a
Analysis		4-Nitrophenol	938
Naval Brass Standards for Optical	C1106		
Emission and Spectroscopic			
Analysis			
Naval Brass Standards for Optical	1 107		
Emission and Spectroscopic			
Analysis			
Naval Brass Standards for Optical	C1107		
Emission and Spectroscopic			
Analysis			

Name	SRM	Name	SRM
Nodular Cast Iron	342a	Organics in Shale Oil	1580
Nominal One Micrometer Polystyrene	1690	Oxalic Acid	4990C
Spheres		Oxygen in Ferrous Materials	1090
Non-Fat Powdered Milk	1549	Ingot Iron	
Nonmagnetic Coating on Magnetic	1359	Oxygen in Ferrous Materials	1091
Substrate (Copper and Chromium		(Stainless Steel AISI 431)	1002
on Steel)		Oxygen in Ferrous Materials Vacuum	1092
Nonmagnetic Coating on Magnetic	1360	Melted Steel Oxygen in Maraging Steel	1094
Substrate (Copper and Chromium		Oxygen in Nitrogen (Gas Standard)	2657
on Steel) Nonmagnetic Coating on Magnetic	1361b	Oxygen in Nitrogen (Gas Standard)	2658
Substrate (Copper and Chromium	13010	Oxygen in Nitrogen (Gas Standard)	2659
on Steel)		Oxygen in Titanium-Base Materials	355
Nonmagnetic Coating on Magnetic	1362a	Oxygen in Valve Steel	1093
Substrate (Copper and Chromium		Oyster Tissue	1566
on Steel)		Palladium, Magnetic Gram	765
Nonmagnetic Coating on Magnetic	1363a	Susceptibility	10.50
Substrate (Copper and Chromium		Penetrant Test Block	1850
on Steel)		Peruvian Soil, Environmental Radioactivity	4355
Nonmagnetic Coating on Magnetic	1364a	Petroleum Crude Oil	1582
Substrate (Copper and Chromium		Phosphate Rock (Florida)	1302 120b
on Steel)	8005	Phosphor Bronze (CDA 521)	871
NPL GM Alpha Alumina NPL GM Alpha Alumina	8006	Phosphor Bronze (CDA 544)	872
NPL GM Alpha Alumina	8007	Phosphorized Copper, Cu VIII	C1251
NPL GM Alpha Alumina	8008	Phosphorized Copper, Cu IX	C1252
NPL GM Graphitized Carbon Black	8001	Phosphorized Copper, Cu X	C1253
NPL GM Graphitized Carbon Black	8002	Phosphorus-32 Radioactivity Standard	4406L-G
NPL GM Melting Point Set	8000	Photographic Step Tablet	1008
NPL GM Non-porous Silica	8003	Pine Needles	1575
NPL GM Non-porous Silica	8004	Plastic Clay	98a
N-tertiary-Butyl-2-benzothiazolesulfen-	384d	Platinum, Magnetic Gram	764
amide Rubber Compound	270	Susceptibility Plutonium-238 Alpha-Particle Standard	4906B
Obsidian Rock	278	Plutonium-240 Alpha-Particle Emission-	4338
Octaphenylcyclotetrasiloxane Oil Furnace Black Rubber Compound	1066a 378b	Rate Solution Standard	4330
Opal Glass Powder	91	Plutonium-239 Alpha-Particle Solution	4331
Optical Emission and X-ray	1102	Standard	
Spectroscopic Analysis		Plutonium-242 Alpha-Particle Solution	4334B
Optical Microscope Linewidth	474	Standard	
Measurement Standard		Plutonium Isotopic Standard	946
Optical Microscope Linewidth	475	Plutonium Isotopic Standard	947
Measurement Standard		Plutonium Isotopic Standard	948
Optical Microscope Linewidth	476	Plutonium Metal	949f 945
Measurement Standard		Plutonium Metal (Standard Matrix Material)	943
		Plutonium-244 Spike Assay and	996
		Isotopic Standard	<i>) ) 0</i>
		Polychlorinated Biphenyls in Oil	1581
		Polycrystalline Alumina Elasticity	718
		Standard	
		Polyester Plastic Film for Oxygen	1470
		Gas Transmission	4.466
		Polyisobutylene Solution in Cetane	1490
		Polystyrene	1478
		Polystyrene (Broad Molecular Weight)	1479 706
		Polystyrene (Broad Molecular Weight) Polystyrene (Narrow Molecular	705
		Weight)	705
		Polystyrene Spheres	1691
		Portland Cement (Black)	1880

Name	SRM	Name	SRM
Portland Cement (Blue)	635	Quartz on Filter Media	2679a
Portland Cement (Clear)	639	Quinine Sulfate Dihydrate	936
Portland Cement (Gold)	634	Radiogenic Lead Isotopic Standard	983
Portland Cement (Green)	638	Radium-226 Gamma-ray Standard	4956
Portland Cement (Pink)	637	Radium-226 Gamma-ray Standard	4957
Portland Cement (Red)	633	Radium-226 Gamma-ray Standard	4958
Portland Cement (White)	1881	Radium-226 Gamma-ray Standard	4959
Portland Cement (Yellow)	636	Radium-226 Gamma-ray Standard	4960
Portland Cement Fineness Standard	114n	Radium-226 Gamma-ray Standard	4961
Potassium Chloride	2202	Radium-226 Gamma-ray Standard	4962
Potassium Chloride (Clinical Standard)	918	Radium-226 Gamma-ray Standard	4963
Potassium Chloride (Primary	999	Radium-226 Gamma-ray Standard	4964B
Chemical)		Radium Standard (Blank Solution)	4952B
Potassium Chloride for Solution	1655	Radon-226 for Radon Analysis	4953C
Calorimetry		Red Brass	1109
Potassium Dichromate	136d	Red Brass	C1109
Potassium Dihydrogen Phosphate	200	Red Brass	1110
Potassium Dihydrogen Phosphate	186Ic	Red Brass	C1110
Potassium Dihydrogen Phosphate	2186I	Red Brass	1111
Potassium Erucate	1076	Red Brass	C1111
Potassium Feldspar	607	Reduced Iron Oxide	691
Potassium Fluoride	2203	Reference Fuel Isooctane	1816a
Potassium Hydrogen Phthalate	185e	Reference Fuel n-Heptane	1815a
Potassium Hydrogen Tartrate	188	Reflection Step Tablet	2061
Potassium Iodide with Attenuator	2033	Refractive Index Glass	1820
Potassium Nitrate	193	Refractive Index Silicone Liquids	1823
Potassium Tetroxalate	189	Refractive Index, Soda-Lime Glass	1822
Powdered Lead Based Paint	1579	Relative Stress-Optical Coefficient	708
Priority Pollutant Polynuclear	1647	Glass	01221
Aromatic Hydrocarbons (in		Resulfurized-Rephosphorized Steel	C1221
Acetonitrile)	16651	Rice Flour	1568
Propane in Air	1665b	River Sediment	1645 4350B
Propane in Air	1666b 1667b	River Sediment, Environmental	4330B
Propane in Air Propane in Air	1668b	Radioactivity Rocky Flats Soil Number 1,	4353
Propane in Air	1669b	Environmental Radioactivity	4000
Propane in Nitrogen (Mobile Source	2643	Rubidium Melting Point	1969
Emission Gas Standard)	2043	Rutile Ore	670
Propane in Nitrogen (Mobile Source	2644	Scanning Electron Microscope	484c
Emission Gas Standard)	2044	Magnification Standard	1010
Propane in Nitrogen (Mobile Source	2645	Scanning Electron Microscope	2069
Emission Gas Standard)	2043	Performance Standard	
Propane in Nitrogen (Mobile Source	2646	Secondary Standard Flexible Disk	3210
Emission Gas Standard)	2010	Cartridge (Computer Amplitude	
Propane in Nitrogen (Mobile Source	2647	Reference)	
Emission Gas Standard)	2017	Secondary Standard Magnetic Tape	3200
Propane in Nitrogen (Mobile Source	2648	Secondary Standard Magnetic Tape	1600
Emission Gas Standard)		Cassette	
Propane in Nitrogen (Mobile Source	2649	Secondary Standard Magnetic Tape	3216
Emission Gas Standard)		Cartridge (Computer Amplitude	
Propane in Nitrogen (Mobile Source	2650	Reference)	
Emission Gas Standard)		Second Surface Aluminum Mirror for	2023
Propane in Nitrogen and Oxygen	2651	Specular Reflectance	
(Mobile Source Emission Gas			
Standard)			
Propane in Nitrogen and Oxygen	2652		
(Mobile Source Emission Gas			
Standard)			
Quartz Cuvette for Spectrophotometry	932		
Quartz for Hydrofluoric Acid	1654		
Solution Calorimetry			

Name	SRM	Name	SRM
Second Surface Aluminum Mirror for	2024	Soda-Lime Sheet Glass	1831
Specular Reflectance		Soda-Lime Silica Glass	622
Second Surface Aluminum Mirror with	2025	Soda-Lime Silica Glass	710
Wedge for Specular Reflectance		Soda-Lime Silica Glass for Liquidus	773
Selenium-Bearing Steel	1170ь	Temperature	
Selenium-75 Radioactivity Standard	4409L-D	Sodium Bicarbonate	191a
Sheet Brass	37E	Sodium Bicarbonate	2191
Silica Brick Silica Brick	198	Sodium Carbonate Sodium Carbonate	192a
Silicon-Aluminum Alloy	199 87a	Sodium Chloride	2192 2201
Silicon Bronze	158A	Sodium Chloride (Clinical Standard)	919
Silicon Density Standard	1840	Sodium Cyclohexanebutyrate	1069b
Silicon Density Standard	1841	Sodium Oxalate Reductometric	40h
Silicon Metal	57a	Standard	
Silicon Powder, Spacing Standard	640a	Sodium Pyruvate	910
for X-ray Diffraction		Sodium Tetraborate Decahydrate	18 <b>7</b> b
Silicon Power Device Level	1522	(Borax)	
Resistivity Standard		Solder	127b
Silicon Resistivity Standard for Eddy	1523	Solder	1131
Current Testers		Special Nuclear Container DOT 6M,	9940
Silver 2-Ethylhexanoate	1077a	15 gal.	0041
Silver-Gold Thermocouple Wire	733	Special Nuclear Container, 55 gal.	9941
Silver, Vapor Pressure	748	Special Nuclear Container Type A, 10 gal.	9942
Sintered and Arc-Cast Tungsten, Thermal Conductivity and	1465	Special Nuclear Container, Type A,	9943
Electrical Resistivity		55 gal.	7743
Sintered and Arc-Cast Tungsten,	1466	Special Nuclear Material Package	9910
Thermal Conductivity and	1400	Spectrographic Ingot Iron and	461
Electrical Resistivity		Low-Alloy Steel Standard (Rod)	
Sintered and Arc-Cast Tungsten,	1467	Spectrographic Ingot Iron and	462
Thermal Conductivity and		Low-Alloy Steel Standard (Rod)	
Electrical Resistivity		Spectrographic Ingot Iron and	463
Sintered and Arc-Cast Tungsten,	1468	Low-Alloy Steel Standard (Rod)	
Thermal Conductivity and		Spectrographic Ingot Iron and	464
Electrical Resistivity	100=	Low-Alloy Steel Standard (Rod)	465
Smoke Density Chamber Standard	1007a	Spectrographic Ingot Iron and	465
(Flaming Exposure Condition) Smoke Density Chamber Standard	1006ь	Low-Alloy Steel Standard (Rod) Spectrographic Ingot Iron and	466
(Non-flaming Exposure Condition)	10000	Low-Alloy Steel Standard (Rod)	400
Soda-Lime Container Glass	621	Spectrographic Ingot Iron and	467
Soda-Lime Flat Glass	620	Low-Alloy Steel Standard (Rod)	107
Soda-Lime Float Glass	1830	Spectrographic Ingot Iron and	468
Soda-Lime Glass	1826	Low-Alloy Steel Standard (Rod)	
Soda-Lime Glass Powder	92	Spectrographic Ingot Iron and	1166
		Low-Alloy Steel Standard	
		Spectrographic Stainless Steel	442
		Standard	
		Spectrographic Stainless Steel	443
		Standard	444
		Spectrographic Stainless Steel	444
		Standard Spectrographic Stainless Steel	D849
		Standard (Disc)	D047
		Spectrographic Stainless Steel	D850
		Standard (Disc)	_ 500
		Spectrographic Stainless Steel	445
		Standard (Group II)	

Name	SRM	Name	SRM
Spectrographic Stainless Steel	446	Spectroscopic Titanium-Base Standard	644
Standard (Group II)		Spectroscopic Titanium-Base Standard	645
Spectrographic Stainless Steel	447	Spectroscopic Titanium-Base Standard	646
Standard (Group II)		Spheroidized Iron Carbide in Ferrite	493
Spectrographic Stainless Steel	448	Spreading Resistance Calibration	2529
Standard (Group II)		(100) n-Type Silicon	
Spectrographic Stainless Steel	449	Spreading Resistance Calibration	2528
Standard (Group II)		(100) p-Type Silicon	
Spectrographic Stainless Steel	450	Spreading Resistance Calibration	2527
Standard (Group II)		(111) n-Type Silicon	
Spectrographic Stainless Steel	849	Spreading Resistance Calibration	2526
Standard (Rod)		(111) p-Type Silicon	
Spectrographic Stainless Steel	850	Stabilized Wine	1590
Standard (Rod)		Stainless Steel	121d
Spectrographic Steel Standard (Disc)	D803a	Stainless Steel	123c
Spectrographic Steel Standard (Disc)	D807a	Stainless Steel	160b
Spectrographic Steel Standard (Rod)	803a	Stainless Steel (AISI 446)	367
Spectrographic Steel Standard (Rod)	804a	Stainless Steel (AISI 446)	1267
Spectrographic Steel Standard (Rod)	805a	Stainless Steel, 13% Chromium	73c
Spectrographic Steel Standard (Rod)	807a	Stainless Steel, Cr-Ni	C1151
Spectrographic Steel Standard (Rod)	808a	Stainless Steel, Cr-Ni	1151a
Spectrographic Steel Standard (Rod)	809a	Stainless Steel, Cr-Ni	C1152
Spectrographic Steel Standard (Rod)	817b	Stainless Steel, Cr-Ni	1152a
Spectrographic Steel Standard (Rod)	820a	Stainless Steel, Cr-Ni	C1153
Spectrographic Steel Standard (Rod)	821	Stainless Steel, Cr-Ni	1153a
Spectrographic Steel Standard (Rod)	827	Stainless Steel, Cr-Ni	C1154
Spectrographic Tool Steel Standard	436	Stainless Steel, Cr-Ni	1154a
Spectrographic Tool Steel Standard	437	Stainless Steel, Cr-Ni-Mo	1155
Spectrographic Tool Steel Standard	438	Stainless Steel, Cr-Ni-Nb	1172
Spectrographic Tool Steel Standard	439	Stainless Steel, Cr-Ni-Ti	1171
Spectrographic Tool Steel Standard	440	Stainless Steel for Pitting or Crevice	1890
Spectrographic Tool Steel Standard	441	Corrosion	730
Spectrographic Tool Steel Standard	837	Stainless Steel Thermal Expansion	738
Spectrographic Tool Steel Standard	840	Stearic Acid Rubber Compound	372h 368
Spectrographic Tool Steel Standard	D837	Steel (AISI 1211)	1169b
(Disc)	W2 0 10	Steel (Lead-Bearing)	1070a
Spectrographic Tool Steel Standard	D840	Strontium Cyclohexanebutyrate	4403L-B
(Disc)	Dout	Strontium-85 Radioactivity Standard	4945D
Spectrographic Tool Steel Standard	D841	Strontium-89 Radioactivity Standard	386h
(Disc)	(25	Styrene-butadiene Rubber (Type 1500)	1970
Spectrographic Zinc-Base Die-Casting	625	Succinonitrile Freezing Point	17c
Alloy A	(2)	Sucrose	2673
Spectrographic Zinc-Base Die-Casting	626	Sulfate and Nitrate on Filter Media	1661a
Alloy B	(27	Sulfur Dioxide in Nitrogen	
Spectrographic Zinc-Base Die-Casting	627	Sulfur Dioxide in Nitrogen	1662a
Alloy C	(20	Sulfur Dioxide in Nitrogen	1663a
Spectrographic Zinc-Base Die-Casting	628	Sulfur Dioxide in Nitrogen	1664a
Alloy D	(20)	Sulfur Dioxide in Nitrogen	1693
Spectrographic Zinc-Base Die-Casting	629	Sulfur Dioxide in Nitrogen	1694
Alloy E	630		
Spectrographic Zinc-Base Die-Casting	030		
Alloy F	621		
Spectrographic Zinc Spelter Standard	631 641		
Spectroscopic Titanium-Base Standard	642		
Spectroscopic Titanium-Base Standard	643		
Spectroscopic Titanium-Base Standard	043		

Name	SRM	Name	SRM
Sulfur Dioxide in Nitrogen	1696	Titanium-Base Alloy (Unalloyed)	650
Sulfur Dioxide Permeation Tube	1627	Titanium-Base Alloy (Unalloyed)	651
(2 cm tube)		Titanium-Base Alloy (Unalloyed)	652
Sulfur Dioxide Permeation Tube	1626	Titanium Dioxide	154b
(5 cm tube)		Toluene	211c
Sulfur Dioxide Permeation Tube	1625	Tomato Leaves	1573
(10 cm tube)	2(02	Tool Steel (AISI M2)	132b
Sulfur in Coal Sulfur in Coal	2682 2683	Tool Steel (AISI M2) Tool Steel Abrasive Wear Standard	1157 1857
Sulfur in Coal	2684	Tracealloy (Nickel-Base	897
Sulfur in Coal	2685	High-Temperature Alloy)	071
Sulfur in Residual Fuel Oil	1619	Tracealloy (Nickel-Base	898
Sulfur in Residual Fuel Oil	1620a	High-Temperature Alloy)	
Sulfur in Residual Fuel Oil	1621b	Tracealloy (Nickel-Base	899
Sulfur in Residual Fuel Oil	1622b	High-Temperature Alloy)	
Sulfur in Residual Fuel Oil	1623a	Trace Elements in a Glass Matrix	610
Sulfur in Residual Fuel Oil	1624a	Trace Elements in a Glass Matrix	611
Sulfur Rubber Compound	371g	Trace Elements in a Glass Matrix	612
Superconductive Thermometric Fixed	767a	Trace Elements in a Glass Matrix	613
Point Device	= 40	Trace Elements in a Glass Matrix	614
Superconductive Thermometric Fixed	768	Trace Elements in a Glass Matrix	615
Point Device	1000-	Trace Elements in a Glass Matrix Trace Elements in a Glass Matrix	616 617
Surface Flammability Standard Synthetic Sapphire	1002c 720	Trace Elements in Coal (Bituminous)	1632a
Technetium-99 Radioactivity Standard	4288	Trace Elements in Coal (Sub-	1635
Technetium-99m Radioactivity	4410H-I	bituminous	1033
Standard	441011-1	Trace Elements in Coal Fly Ash	1633a
Tetrachloroethylene in Nitrogen	1808	Trace Elements in Fuel Oil	1634a
Thallium-201 Radioactivity Standard	4404L-F	Trace Elements in Water	1643a
Thermal Resistance, Fibrous Glass	1451	Trace Mercury in Coal	1630
Batt		2,2,4-Trimethylpentane	217c
Thermal Resistance, Fibrous Glass	1450b	Tripalmitin	1595
Board		Tris, Basimetric	723a
Thorium-228, Thallium-208 Gamma-ray	4206C	Tris, for Solution Calorimetry	724a
Point-Source Standard	6470	Tris(hydroxymethyl)aminomethane	922
Tin-Base Bearing Metal Tin, Freezing Point	54D 741	Tris(hydroxymethyl)aminomethane hydrochloride	923
Tin-113-Indium-113m Radioactivity	4402L·C	Tris(1-phenyl-1, 3-butanediono)	1078b
Standard	4402L-C	Chromium (III)	10780
Tin-121m Point-Source Gamma-ray	4264B	Tris(1-phenyl-1, 3-butanediono)	1079ь
Emission-Rate Standard	42~	Iron (III)	1071b
Tin, Secondary Freezing Point Standard	42g	Triphenyl Phosphate Tungsten Carbide	2-4
Titanium Alloy	654a	Tungsten-Chromium-Vanadium Steel	276a 50c
Titanium-Base Alloy	173b	Tungsten Concentrate	277
Titanium-Base Alloy	176	Tungsten, Heat Capacity	782
,		Tungsten-20% Molybdenum Alloy	480
		Electron Microprobe Standard	
		Tungsten Thermal Expansion	737
		Unalloyed Copper	1034
		Unalloyed Copper, Cu "O"	393
		Unalloyed Copper, Cu IV	457
		Unalloyed Copper, Cu XI	454
		Unalloyed Copper, Cu I (Chip)	394
		Unalloyed Copper, Cu II (Chip)	395 396
		Unalloyed Copper, Cu III (Chip) Unalloyed Copper, Cu V (Chip)	398
		Unalloyed Copper, Cu VI (Chip)	399
		Unalloyed Copper, Cu VII (Chip)	400
		Unalloyed Copper, Cu I (Rod)	494
		- •	

Name	SRM	Name	SRM
Unalloyed Copper, Cu II (Rod)	495	Wear-Metals in Lubricating Oil	1085
Unalloyed Copper, Cu III (Rod)	496	(300 ppm)	
Unalloyed Copper, Cu V (Rod)	498	Wheat Flour	1567
Unalloyed Copper, Cu VI (Rod)	499	White Cast Iron	338
Unalloyed Copper, Cu VII (Rod)	500	White Cast Iron (Disc)	1145
Unalloyed Titanium	354	White Cast Iron (Disc)	1146
Uranium Isotopic Standard (Nominally	U-0002	White Cast Iron (Disc)	1150
depleted to 0.02%)	0 0002	White Ceramic Tile for Directional	2019b
Uranium Isotopic Standard	U-005a	Hemispherical Reflectance	20170
Uranium Isotopic Standard	U-010	White Ceramic Tile for Directional	2020
(Nominally 1% Enriched)	0.010	Hemispherical Reflectance	2020
Uranium Isotopic Standard	U-015	White Iron	3d
(Nominally 1.5% Enriched)	0-013	White Opan Glass Diffuse Spectral	2015
	U-020	Reflectance Standard for the	2015
Uranium Isotopic Standard	U-030a		
Uranium Isotopic Standard	U-050	Visible Spectrum	4309G
Uranium Isotopic Standard	0-030	Xenon-127 Gaseous Radioactivity	4309G
(Nominally 5% Enriched)	11100	Standard	42071
Uranium Isotopic Standard	U-100	Xenon-133 Gaseous Radioactivity	4307I
(Nominally 10% Enriched)	11.150	Standard	
Uranium Isotopic Standard	U-150	Xenon-133 Gaseous Radioactivity	4415L-I
(Nominally 15% Enriched)		Standard	
Uranium Isotopic Standard	U-200	Xenon-133, Xenon-137, Krypton-85	4310B
(Nominally 20% Enriched)		Mixed Gaseous Radioactivity	
Uranium Isotopic Standard	U-350	Standard	
(Nominally 35% Enriched)		X-ray Film Step Tablet	1001
Uranium Isotopic Standard	U-500	X-ray Powder Diffraction Intensity	674
(Nominally 50% Enriched)		Standard	
Uranium Isotopic Standard	U-750	X-ray Powder Diffraction (Mica)	675
(Nominally 75% Enriched)		Low 2 Theta	
Uranium Isotopic Standard	U-800	Ytterbium-169 Radioactivity Standard	4419L-E
(Nominally 80% Enriched)		Zinc-Base Alloy (Die Casting)	94c
Uranium Isotopic Standard	U-850	Zinc Concentrates	113a
(Nominally 85% Enriched)		Zinc Concentrates	329
Uranium Isotopic Standard	U-900	Zinc Cyclohexanebutyrate	1073b
(Nominally 90% Enriched)		Zinc, Freezing Point	740
Uranium Isotopic Standard	U-930	Zinc, Freezing Point Standard	43h
(Nominally 93% Enriched)		Zinc Metal	683
Uranium Isotopic Standard	U-970	Zinc Oxide Rubber Compound	370e
(Nominally 97% Enriched)		Zircaloy-2	360a
Uranium Metal	960	Zircaloy-4 Metal	1237
Uranium Oxide	950b	Zircaloy-4 Metal	1238
Uranium Oxide	969	Zircaloy-4 Metal	1239
Uranium-233 Spike Assay and	995	Zirconium-Barium Chromate	1651
Isotopic Solution Standard		Formulation for Heat-Source	
Uranium-235 Spike Assay and	993	Powder Calorimetry	
Isotopic Solution Standard		Zirconium-Barium Chromate	1652
Urban Dust/Organics	1649	Formulation for Heat-Source	
Urban Particulate Matter	1648	Powder Calorimetry	
Urea	912a	Zirconium-Barium Chromate	1653
Urea	2141	Formulation for Heat-Source	1000
_	2152	Powder Calorimetry	
Urea	913	Zirconium Metal	1234
Uric Acid Vanadium and Nickel in Residual	1618	Zirconium Metal Zirconium Metal	1234
Vanadium and Nickel in Residual	1010	Zirconium Metal	1235
Fuel Oil	8505	Zircomuni wietai	1230
Vanadium in Curde Oil	4266		
Vanadium-49 Low-Energy Photon	4200		
Standard	349		
Waspaloy	1084		
Wear-Metals in Lubricating Oil (100 ppm)	1004		

U. S. Department of Commerce Frederick B. Dent Secretary

National Bureau of Standards Richard W. Roberts, Director Appendix II. Certificates for the Environmental Research, Analysis, and Control Standards (listed in numerical order).

## National Bureau of Standards Certificate of Analysis Standard Reference Material 1579

#### Powdered Lead Based Paint

This Standard Reference Material is intended for use in the calibration of apparatus and methods used in the determination of lead in paint removed from the interior surfaces of old housing. The certified value is based on at least a 100 milligram sample of the as-received, total material.

The certified value of 11.87 percent lead is the weighted average value determined by a statistical analysis of the results of 32 determinations by atomic absorption spectrometry (average 11.84 percent lead, s=0.13 percent lead), and 16 determinations by polarography (average 11.93 percent lead, s=0.13 percent lead). The standard error of the weighted average is 0.02 percent lead, and the half-width of the 95 percent confidence interval is taken to include  $\pm 0.04$  percent lead by weight.

X-ray fluorescence spectrometry showed the bottle-to-bottle inhomogeneity of the material with respect to lead content to be no greater than 0.02 percent lead; no within-bottle inhomogeneity was detected.

Analyses for lead and determinations of homogeneity were carried out in the NBS Analytical Chemistry Division by the following persons:

X-ray Fluorescence: S. D. Rasberry

Atomic Absorption Spectrometry: T. C. Rains and T. A. Rush

Polarography: E. J. Maienthal

Statistical calculations were carried out by J. Mandel of the NBS Institute for Materials Research.

The overall direction and coordination of the technical measurements leading to this certificate were performed under the chairmanship of B. Greifer.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Washington, D. C. 20234 January 23, 1973 J. Paul Cali, Chief Office of Standard Reference Materials

#### Preparation, Testing, and Analysis

#### Collection

The paint for this Standard Reference Material was collected by the staff of the Philadelphia Department of Public Health from the interior surfaces of dwellings undergoing renovation. The paint was softened with a hand torch, scraped from the plaster and wood substrates, and collected in plastic bags as a heterogeneous mixture of many different kinds of paints. In the laboratory, non-paint matter such as bits of metal, plastic, glass, and wood were removed and the paint mixture was ground in a disk mill to produce a material suitable for feeding into a jet mill. The paint was comminuted in a jet mill operating at 100 psig air pressure, then sieved through a 100-mesh vibrating screen to remove the coarse, non-grindable fraction. Two additional passes through the jet mill at 97 to 107 psig gave a fine powder with 99.31 weight percent passing through a 325 mesh sieve.

#### Homogeneity

Sample homogeneity was ascertained by x-ray fluorescence analysis for lead content on 17 samples chosen at random from the total lot. A statistical analysis of the data from 136 observations showed the bottle-to-bottle variability among the samples to be no greater than 0.02 percent lead. No within-bottle variation with respect to lead was detected.

#### Dissolution

A procedure used to dissolve the sample is summarized briefly: dry ash the weighed paint for 2 hours at 450 °C, digest with 2:5 HC1 - HNO3 containing HF, evaporate to dryness; treat with HNO3, evaporate to dryness; treat twice with HCl and evaporate to dryness each time. Extract the solids twice with portions of acetic acid - ammonium acetate solution, heating for several hours just below boiling. Combine the extracts and heat the mixture (including solids) for one hour, just below boiling. Cool the mixture and determine lead in solution. (The solids need not be removed for polarographic analysis.)

An alternate procedure for sample dissolution is: dry ash the weighed paint for 6 hours at 500 °C, cool, then digest for 2 hours in 1:1 HCl-HNO<sub>3</sub>. Separate the insoluble solids from the solution by centrifuging, and wash 3 times with 1:10 HNO<sub>3</sub> combining the rinsings with the principal solution. Determine lead in solution.

Details of the dissolution procedures, the analytical procedures, and results will be published in the 260 series of NBS Special Publications.

U. S. Department of Commerce Philip M. Klutznick Secretary National Survey of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis Standard Reference Material 1580 Organics in Shale Oil

This SRM is intended primarily for evaluating the reliability of analytical methods for the determination of trace level organic compounds in an oil matrix, i.e., shale oil, petroleum crude oil, or coal-derived liquids.

Certified Values of Constituent Organic Compounds: The certified values for selected organic constituents are shown in Table 1. These values are based on results obtained by two independent, analytical methods (see Table 2). Non-certified values, which are given for information only, are listed in Table 3.

#### NOTICE AND WARNINGS TO USER

Expiration of Certification: This certification is valid, within the limits certified, for 3 years from the date of purchase. In the event that the certification should become invalid before then, purchasers will be notified by NBS.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures between 10-30 °C.

Use: Samples for analysis should be withdrawn from ampoules immediately after opening and processed without delay for any certified value in Table 1 to be valid within the stated uncertainty. Certified values are not applicable to ampoules stored after opening, even if resealed.

#### PREPARATION AND ANALYSIS

The shale oil for this SRM came from a 150-ton retort for *in-situ* simulated combustion of oil shale, operated by the Laramie Energy Technology Center, Laramie, Wyoming. The shale was from the Mahogany Zone of the Colorado Green River Formation. The shale oil had been supplied in November 1975 to the Oak Ridge National Laboratory (ORNL) where it underwent centrifugation to separate the oil from water and sludge. The shale oil was provided to NBS by Bruce R. Clark, ORNL, Oak Ridge, Tennessee.

At NBS, the centrifuged sample was filtered through fine filter paper and mixed in a 20-liter, Teflon-stoppered, glass bottle by rolling for 40 hours. Samples were aliquoted into 2-mL amber glass ampoules. Although not intended to be representative of all shale oils, SRM 1580 provides a typical specimen of this matrix for use in developing analytical methods.

Randomly selected ampoules were analyzed. Each analyst examined at least six ampoules, sometimes measuring replicates from one ampoule. No trend was found in measured values with the ampouling sequence.

Two independent techniques were employed for the determination of the certified values for the organic constituents. Three different methods of sample preparation were used prior to analysis: simple dilution of the shale oil with methylene chloride (or other suitable solvent); acid/base extraction to isolate acidic, basic, and neutral components; and a high performance liquid chromatographic fractionation. The following techniques were employed for the final quantitative analysis: gas chromatography (GC), gas chromatography/mass-spectrometry (GC/MS) with single ion monitoring for selective detection, and high performance liquid chromatography (HPLC) with selective fluorescence detection. All GC/MS analyses used the standard addition method for quantitation. The GC and HPLC analyses employed either internal standard, external standard, or standard addition methods. The analytical methods and the corresponding values are summarized in Table 2.

Consultation on the statistical design of the experimental work was provided by K. R. Eberhardt of the Statistical Engineering Division.

The coordination of the technical measurements leading to certification were performed under the direction of H. S. Hertz, S. N. Chesler, L. R. Hilpert, W. E. May, and S. A. Wise.

The technical and support aspects involved in preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234 November 24, 1980 (Revision of Certificate dated 3-10-80)

(over)

George A. Uriano, Chief Office of Standard Reference Materials The following members of the staff of the Center for Analytical Chemistry, Organic Analytical Research Division, performed the analytical determinations.

1. J. M. Brown-Thomas 5. P. L. Konash 2. S. N. Chesler 6. W. E. May 3. F. R. Guenther 7. R. M. Parris 4. L. R. Hilpert 8. K. L. Richie

TABLE 1. Certified Values of Organic Constituents

Compound	Concentration $(\mu g/g^a)$
Fluoranthene	54 ± 10
Pyrene	$104 \pm 18$
Benzo[a]pyrene	$21 \pm 6$
Benzo[e]pyrene	18 ± 8
Perylene	$3.4 \pm 2.2$
Phenol	407 ± 50
o-Cresol	$385 \pm 50$
2,6-Dimethylphenol	$175 \pm 30$
Benzo[f]quinoline	16 ± 4
(5,6-Benzoquinoline)	

<sup>a</sup>The estimated uncertainty listed for a constituent is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material inhomogeneity. The estimated uncertainty is intended to correspond to approximately 95% confidence limits.

TABLE 2. Summary of Results by the Analytical Methods Used in Certification

		Number of Ampoules	Sample Preparation	Analytical
Compound	Concentration $(\mu g/g)^a$	Analyzed	Technique	Technique
Fluoranthene	55 ± 5	6	Direct Injection	GC/MS
	53 ± 2	9	HPLC	HPLC
Pyrene	$101 \pm 5$	6	Direct Injection	GC/MS
	107 ± 8	10	HPLC	HPLC
Benzo[a]pyrene	$20 \pm 1$	6	Direct Injection	GC/MS
	23 ± 1	8	HPLC	HPLC
Benzo[e]pyrene	17 ± 1	6	Direct Injection	GC/MS
	$20 \pm 3$	8	HPLC	HPLC
Perylene	$2.8 \pm 0.6$	5	Direct Injection	GC/MS
	$3.9 \pm 0.6$	11	HPLC	HPLC
Phenol	$412 \pm 35$	8	HPLC	GC/MS
	$402 \pm 4$	8	Acid/Base Extraction	GC
o-Cresol	$386 \pm 42$	8	HPLC	GC/MS
	$384 \pm 9$	8	Acid/Base Extraction	GC
2,6-Dimethylphenol	$183 \pm 23$	9	HPLC	GC/MS
	$168 \pm 8$	8	Acid/Base Extraction	GC
Benzo[f]quinoline	$16 \pm 1$	7	HPLC	HPLC
(5,6-Benzoquinoline)	$15 \pm 1$ rd deviation of a single measuren	8	Acid/Base Extraction	Multi- dimen- sional GC
Uncertainty is the standar	ia aeviation of a single measuren	ICIII.		UC

<sup>&</sup>lt;sup>a</sup>Uncertainty is the standard deviation of a single measurement.

#### TABLE 3. Non-Certified Values of Organic Compounds in Shale Oil

NOTE: The values shown in this table are not certified because they are not based on the results of two independent methods. These values are included for information only.

Compound	Concentration $(\mu g/g)$
p-Cresol	$(270)^a$
m-Cresol	$(330)^a$
2,5-Dimethylphenol	$(320)^{a}$
2,4-Dimethylphenol	$(380)^{a}$
2,5,6-Trimethylphenol	(360) <sup>a</sup>
2,4,6-Trimethylphenol	(120) <sup>a</sup>
Phenanthridine <sup>a</sup> Acid/base extraction - GC analysis	(45) <sup>b</sup>

<sup>&</sup>quot;Acid/base extraction - GC analysis bHPLC extraction - HPLC analysis

U. S. Department of Commerce Malcolm Baldrige Secretary National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis

#### Standard Reference Material 1620a

#### Sulfur in Residual Fuel Oil

Sulfur Concentration . . . . . 4.504 ± 0.010 weight percent

This Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1620a is a commercial "No. 5 Heavy" residual fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certified using three independent methods of analysis: gravimetry, ion chromatography, and x-ray fluorescence.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE AND RECOMMENDED USE: Due to the high sulfur content of SRM 1620a, it is recommended that the bottle be shaken vigorously before sampling. Homogeneity and stability testing at NBS indicates that the best results are achieved when the material is shaken before use.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1620a was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

<u>Table 1</u>
Physical Properties for SRM 1620a

Flash Point " °C	Kinematic Viscosity <sup>b</sup> 50 °C (cSt)	Pour Point <sup>c</sup> °C	Density @ 20 ° C <sup>d</sup> g/cm <sup>3</sup>
70	47.75	2	1.096

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

#### Methods Used for Physical Tests

- a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester
- b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids
- c. ASTM D97-66 (1978) Pour Point of Petroleum Oils
- d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

<u>Table 2</u>

<u>Semi-Quantitative Emission Spectrometry</u>

Analysis for SRM 1620a

Element	μg/ mL	Elemen	t μg, mL
Al	20	Mo	<1
B	<1	Na	31
Ca	9	Ni	<1
Cr	<1	Si	13
Cu	<1	Sn	<1
Fe	<5	Ti	<1
Mg	<1	V	<1
Mn	<1	Zn	23

Note: SRM 1620a was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed by J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry.

U. S. Department of Commerce Malcolm Baldrige Secretary National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis

#### Standard Reference Material 1621b

#### Sulfur in Residual Fuel Oil

Sulfur Concentration . . . .  $0.950 \pm 0.005$  weight percent

This Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1621b is a commercial "No. 6" residual fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certified using three independent methods of analysis: gravimetry, ion chromatography, and x-ray fluorescence.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE: The certification of SRM 1621b is valid for 3 years from date of purchase.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1621b was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

Table 1
Physical Properties for SRM 1621b

Flash Point <sup>a</sup> °C	Kinematic Viscosity <sup>b</sup> 50 °C (cSt)	Pour Point <sup>c</sup> °C	Density @ 20 °C <sup>d</sup> g/cm <sup>3</sup>
111	89.2	11	0.929

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

#### Methods Used for Physical Tests

- a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester
- b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids
- c. ASTM D97-66 (1978) Pour Point of Petroleum Oils
- d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

Table 2
Semi-Quantitative Emission Spectrometry
Analysis for SRM 1621b

Element	μg/mL	Element	μg/mL
Al	6	Мо	<1
В	<1	Na	8
Ca	9	Ni	6
Cr	3	Si	6
Cu	<1	Sn	<1
Fe	<5	Ti	<1
Mg	<1	V	15
Mn	i	Zn	15

Note: SRM 1621b was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed by J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry.

U. S. Department of Commerce Malcolm-Baldrige Secretary National Bureau of Standards Ernest Ambler, Director

## National Bureau of Standards

### Certificate of Analysis

#### Standard Reference Material 1622b

#### Sulfur in Residual Fuel Oil

Sulfur Concentration . . . . . 1.982 ± 0.018 weight percent

This Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1622b is a commercial "No. 6" residual fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certified using three independent methods of analysis: gravimetry, ion chromatography, and x-ray fluorescence.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE: The certification of SRM 1622b is valid for 3 years from date of purchase.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1622b was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

Table 1
Physical Properties for SRM 1622b

Flash Point <sup>a</sup> °C	Kinematic Viscosity <sup>b</sup> 50 °C (cSt)	Pour Point <sup>c</sup> °C	Density @ 20 °C <sup>d</sup> g/cm <sup>3</sup>
65	377.34	-7	0.984

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

#### Methods Used for Physical Tests

- a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester
- b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids
- c. ASTM D97-66 (1978) Pour Point of Petroleum Oils
- d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

Table 2
Semi-Quantitative Emission Spectrometry
Analysis for SRM 1622b

Element	μg/mL	Element	μg/ mL
Al	8	Мо	<1
В	<1	Na	25
Ca	24	Ni	15
Сг	1	Si	13
Cu	<1	Sn	<1
Fe	<5	Ti	<1
Mg	2	V	50
Mn	1	Zn	11

Note: SRM 1622b was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry

U. S. Department of Commerce Malcolm Baldrige Secretary

National Bureau of Standards Frnest Ambler, Director

## National Bureau of Standards

### Certificate of Analysis

#### Standard Reference Material 1623a

#### Sulfur in Residual Fuel Oil

Sull'ur Concentration. . . . . . 0.240 ± 0.003 weight percent

This Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1623a is a commercial "No. 5 Heavy" residual fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certified using three independent methods of analysis: gravimetry, ion chromatography, and x-ray fluorescence.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE: The certification of SRM 1623a is valid for 3 years from date of purchase.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1623a was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

<u>Table 1</u>
Physical Properties for SRM 1623a

Flash Point " °C	Kinematic Viscosity <sup>h</sup> 50 °C (cSt)	Pour Point <sup>c</sup> °C	Density @ 20 °C <sup>d</sup> g/cm <sup>3</sup>
140	53.82	17	0.918

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

#### Methods Used for Physical Tests

- a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester
- b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids
- c. ASTM D97-66 (1978) Pour Point of Petroleum Oils
- d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

<u>Table 2</u>

<u>Semi-Quantitative Emission Spectrometry</u>

Analysis for SRM 1623a

Element	μg/ mL	Element	μg/mL
Al	5	Мо	<1
В	<1	Na	9
Ca	9	Ni	1
Cr	1	Si	<1
Cu	<1	Sn	<1
Fe	<5	Ti	<1
Mg	<1	V	3
Mn	<1	Zn	15

Note: SRM 1623a was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed by J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry.

U. S. Department of Commerce Malcolm Buldrige Secretary National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis

#### Standard Reference Material 1624a

Sulfur in Distillate (Diesel) Fuel Oil

Sulfur Concentration . . . . . 0.141 ± 0.002 weight percent

This Standard Reference Material is intended for use as an analytical standard in the determination of total sulfur in fuel oils or materials of similar matrices. SRM 1624a is a commercial "No. 2-D" distillate fuel oil as defined by American Society for Testing and Materials, ASTM.

Sulfur was certified using two independent methods of analysis: gravimetry and ion chromatography.

The standard error of the certified value includes observed variability within and between measurement methods and any observed material heterogeneity.

NOTICE: The certification of SRM 1624a is valid for 3 years from date of purchase.

Analyses for certification were performed by W. F. Koch and E. R. Deardorff of the Inorganic Analytical Research Division and P. A. Pella of the Gas and Particulate Science Division.

The statistical analysis of the certification data was performed by R. C. Paule of the National Measurement Laboratory.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 December 22, 1981 George A. Uriano, Chief Office of Standard Reference Materials

SRM 1624a was also tested and found to exhibit the physical properties shown in Table 1. In addition, semi-quantitative values obtained by emission spectrometry are given in Table 2. These values are not certified, but supplied for information only.

<u>Table 1</u>
Physical Properties for SRM 1624a

Flash Point a	Kinematic Viscosity <sup>b</sup> 40 °C (cSt)	Cloud Point <sup>c</sup> °C	Density @ 20 °C <sup>d</sup> g/cm <sup>3</sup>
53	2.57	-14	0.848

These measurements were performed by S. Weeks, Materials Chemistry Division, Center for Material Science.

#### Methods Used for Physical Tests

- a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester
- b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids
- c. ASTM D2500-66 (1976) Cloud Point of Petroleum Oils
- d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

Table 2
Semi-Quantitative Emission Spectrometry
Analysis for SRM 1624a

Element	μg/mL	Element	$\mu g/mL$
Al	1	Мо	<1
В	<1	Na	<1
Ca	7	Ni	<1
Cr	<1	Si	<1
Cu	<1	Sn	<1
Fe	<5	Ti	<1
Мg	<1	V	<1
Mn	<1	Zn	<1

Note: SRM 1624a was analyzed using the rotating disc method. This method is based on absolute amounts of sample since no internal standard is used to correct for the amount of sample actually analyzed. Differences in actual values may range from factors of 1-3.

These measurements were performed by J. A. Norris, Inorganic Analytical Research Division, Center for Analytical Chemistry.

U.S. Department of Commerce Juanita M. Kreps Secretary

National Bureau of Standards Ernest Ambler, Director

### National Bureau of Standards

### Certificate of Analysis

#### Standard Reference Material 1630

#### Trace Mercury in Coal

This Standard Reference Material is intended as an analytical standard for the determination of trace mercury in coal. The material is a commercially available coal that was crushed to a size of 210 to 500 micrometers with a roll crusher. From a total of 500 packaged bottles, 30 were randomly selected for analysis. Duplicate determinations were made on 0.5 g portions of 25 of these bottles, and single determinations were made on the other five. The mercury content of this material was obtained by destructive neutron activation analysis.

The recommended value is the average of these 55 determinations on 30 bottles, which was found to be:

Mercury content =  $0.13 \mu g/g$ 

The recommended value is not expected to change by more than ± 1 in the last significant figure.

A study of homogeneity showed no variability among bottles that could not be accounted for by analytical error. Duplicate samples from the same bottle indicated a homogeneity for mercury of  $\pm$  5% (relative).

The mercury content was also determined by flameless atomic absorption spectrometry, yielding an average value of  $0.14 \mu g/g$ .

Selenium was also determined using destructive neutron activation analysis. The value obtained, which is not certified but included for information only, was found to be  $2.1 \mu g/g$ .

The homogeneity testing and analyses for certification were performed in the NBS Analytical Chemistry Division by T. E. Gills and H. Rook under the direction of P. D. LaFleur.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Materials were coordinated through the Office of Standard Reference Materials by C. L. Stanley.

Washington, D.C. 20234 August 1, 1979 (Revision of Certificate dated 11-2-71 Editorial Revision only.) George A. Uriano, Chief Office of Standard Reference Materials

#### ANALYTICAL PROCEDURE

The bottles containing the samples were allowed to remain open at room temperature (about 25 °C) for twenty-four hours.

The coal samples, along with solution standards of mercury and NBS Standard Reference Material 1571 (Orchard Leaves) used as a control, were encapsulated in cleaned quartz vials. The geometry of both the samples and the standards were optimized so that flux monitors were not needed. The samples were irradiated for four hours at a thermal flux of  $6 \times 10^{13} \, \text{ n·cm}^{-2} \, \text{sec}^{-1}$ . The samples were allowed to decay for three days to minimize the personnel dose rate. The samples were postweighed into porcelain boats and burned in a combustion tube. The volatile mercury compounds and other volatile products liberated during burning were trapped in a liquid nitrogen cold trap. The cold trap was allowed to warm to room temperature. The mercury compounds were then transferred to polyethylene bottles by washing the cold trap with concentrated nitric acid and water. For this analysis,  $^{197}$ Hg produced by  $^{196}$ Hg(n,  $\gamma$ )  $^{197}$ Hg was used as the measuring activity.

Bromine-82, an interfering isotope, was separated from the sample by using the classical silver bromide precipitation.

The samples were counted on a 22 cm<sup>3</sup> Ge(Li) detector connected to a 2048-multichannel analyzer. The accumulated data was processed by computer for peak identification and integration. The concentrations were determined by using a Standard Comparator Method.

#### NOTE TO USER

It is suggested that persons using SRM 1630 to check their analytical technique should adopt the following criteria. If the average,  $\overline{X}$ , of N replicate measurements on this SRM is found to lie in the interval—

$$0.127 - \frac{0.013}{\sqrt{N}} < \vec{X} < 0.127 + \frac{0.013}{\sqrt{N}}$$

then the analytical technique used gives a result compatible with that found at NBS. However, if the value  $\overline{X}$  lies outside this interval, then the technique should be examined for possible bias or miscalibration.

NOTE: The above expression is not rigorously correct. It does not include a possible component for between laboratory variability nor sources of systematic error.

U. S. Department of Commerce Malcolm Baldrige Secretary National Burgeu of Standards Ernest Ambler, Director

## National Bureau of Standards

### Certificate of Analysis

#### Standard Reference Material 1632b

Trace Elements in Coal

(Bituminous)

This Standard Reference Material (SRM) is intended for use in the calibration of apparatus and the evaluation of techniques employed in the analysis of coal or similar materials. SRM 1632b is a bituminous coal with a nominal sulfur content of 1.9%. It is in the form of a fine powder (-60 mesh).

Certified Values of Constituent Elements: The certified values for the constituent elements are given in Table 1. The certified values are based on measurements using proven techniques and methods. Noncertified values are given in Table 2 and are provided for information only. These values are based on measurements made using a single technique or method. While no reason exists to suspect systematic bias in the information values, no attempt was made to determine if such a bias exists that is attributable to the technique and/or method used. A list of analytical techniques and methods used for the different analyses is given in Table 3. As part of its update certification program, NBS will periodically update many of these values to certification status.

Expiration of Certification: The certification of SRM 1632b will be valid up to 5 years from the purchase date. Should any of the certified constituents become invalid prior to that date, purchasers will be notified by NBS.

<u>Use:</u> This material should be vacuum dried at ambient temperature for 24 hours prior to use. The certified concentrations are reported on a "dry-weight" basis, thus the concentration determined on undried samples should be adjusted for the moisture content of the sample. Typical moisture loss using the drying procedure stated above is 1.3%.

A minimum sample size of 250 mg of the dried material is required for the certified values to be valid.

This SRM should be kept in its original bottle. It should not be exposed to intense source of radiation, including ultraviolet lamps or sunlight.

The statistical analysis of the certification data was performed by R.C. Paule of the National Measurement Laboratory.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Gaithersburg, MD 20899 June 20, 1985 Stanley D. Rasberry, Chief Office of Standard Reference Materials

Source and Preparation of Material: The coal for this SRM was obtained from the Humphrey No. 7 mine and coal preparation plant of the Consolidation Coal Company, Christopher Coal Company Division, Osage, West Virginia. This mine produces bituminous coal with a sulfur content of 1.8-1.9 percent (dry basis). This coal was obtained from an underground mine that recovers coal from the Pittsburgh seam, which is considered the single most valuable and extensive coal seam in the United States.

Approximately 900 kg of the coal for SRM 1632b was oven dried prior to processing, in accordance with procedures outlined in ASTM D2013. The coal was reduced in size to -60 mesh and sieved prior to blending. The coal was then blended in a stainless steel cone blender (approximate capacity 0.85 cubic meter). After blending the coal was packaged in polyethylene-lined aluminum cans and was subsequently repackaged in fifty gram units.

#### Analysis

Major, Minor, and Trace Constituents: In general, the major, minor, and trace constituents were certified using two or more independent methods of analysis or two or more different laboratories. For those constituents that were determined using a single method, technique, or laboratory, the values are given for information only. (See Table 3).

Calorific Value: The calorific value was determined using measurements made in an isoperibol calorimeter, an isothermal calorimeter, and an adiabatic calorimeter at two different laboratories.

Moisture, Ash, and Volatile Matter: The moisture, ash, and volatile matter values were determined on measurements made using the standard ASTM methods, D3173, D3174, and D3175, respectively. In addition, commercial instruments commonly used for the determination of the parameters provided additional values.

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Table 1. Certified Values of Constituent Elements

Major Constituents		Minor	Constituents
Content			Content
Elements	Wt. Percent	Elements	Wt. Percent
Carbon (Total)	$78.11 \pm 0.37^{a}$	Aluminum	$0.855 \pm 0.019$
Hydrogen	$5.07 \pm 0.06$	Calcium	$0.204 \pm 0.006$
Nitrogen	$1.56 \pm 0.07$	Iron	$0.759 \pm 0.045$
Sulfur	$1.89 \pm 0.06$	Magnesium	$0.0383 \pm 0.0008$
Volatile matter	35.4 ± 1.1	Potassium	$0.0748 \pm 0.0028$
		Sodium	$0.0515 \pm 0.0011$
		Titanium	$0.0454 \pm 0.0017$

#### Trace Constituents

Element	Conten µg/g	ıt	Element		ntent g/g
Arsenic	$3.72 \pm 0$	.09	Manganese	12.4	±1.0
Barium	67.5 ±2	.1	Nickel	6.10	$\pm 0.27$
Cadmium	$0.0573 \pm 0$	.0027	Rubidium	5.05	$\pm 0.11$
Cobalt	$2.29 \pm 0$	.17	Selenium	1.29	$\pm 0.11$
Copper	$6.28 \pm 0$	.30	Thorium	1.342	$2 \pm 0.036$
Lead	$3.67 \pm 0$	.26	Uranium	0.436	$5 \pm 0.012$
			Zinc	11.89	$\pm 0.78$

Calorific Value <sup>b, c</sup>	Ash, wt.%
$14005 \pm 35 \text{ Btu/lb} (32.57 \pm 0.08 \text{ MJ kg}^{-1})$	$6.79 \pm 0.16$

<sup>&</sup>lt;sup>a</sup> The listed ±uncertainties for carbon, hydrogen, volatile matter, and calorific value are two standard deviations of the certified value. The listed ± uncertainties for all other constituents are two standard deviations for the certified values and include an allowance for minor sample heterogeneity. The observed sample variability was generally less than two percent of the constituent value.

Table 2. Noncertified Values for Constituent Elements

	Trace Co	nstituents	
Element	Content µg/g	Element	Content μg/g
Antimony	(0.24)	Lithium	(10)
Bromine	(17)	Molybdenum	(0.9)
Cerium	(9)	Samarium	(0.87)
Cesium	(0.44)	Scandium	(1.9)
Chlorine	(1260)	Silicon, wt %	(1.4)
Chromium	(11)	Strontium	(102)
Europium	(0.17)	Tungsten	(0.48)
Hafnium	(0.43)	Vanadium	(14)
Lanthanum	(5.1)		

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<sup>&</sup>lt;sup>b</sup>The calorific value (MJ kg<sup>-1</sup>) may decrease upon aging or normal oxidation of the coals. NBS will continue to monitor this value and report any substantive change in the certified calorific value to the purchaser. The reference date for the calorific value is May 1985.

<sup>&</sup>lt;sup>c</sup>The calorific value is determined as HHV2 (Higher Heating Value-Moisture Free).

Table 3. Analytical Techniques and Methods Used for the Characterization of SRM 1632b

Method/ Element	A	В	С	D	E	F	G	Н	1	J	К	L	М
Al			•	•								•	
As			•		•								
Ash Content							• 2		•				
Ва			•										
Br			•										
C (Total)							• 5	•	•		•		
Ca		•	•	•								•	
Cal Val									•	•			
Cd	•	•							ļ			-	<b>†</b>
Ce			•			<b></b>	<del> </del>						
Cl			•										
Со			•	•									
Cr			•				1	-					•
Cs			•										-
Cu					•	-							•
Eu			•	-	-	-						-	
Fe	•		•		-	1						•	-
		-					- 6	•					-
H			•		-	-	₹ 5		•			-	-
Hſ		•			-	-						-	-
K		•	•	•								•	-
La			•		-		ļ		ļ			-	
Li				•									ļ
Mg	•	•	•			-							-
Mn		-	•	•	-								-
Мо			•	ļ		ļ							ļ
N							• 6					ļ	
Na			•	•							L	<u> </u>	
Ni	•												•
Pb	•	•											
Rb		•	•	•								1	
S						•	• 4		•			•	
Sb			•										
Sc			•										
Se			•		•								
Si			٠									•	
Sm			•										
Sr			•										
Th		•	•										
Ti			•	•								•	•
U		•	•										
V			•										•
Volatile Matter							• 3		•				-
· Jiaciic Mattel			-						-	-		-	
W			•						1				

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#### Analytical Methods

- A. Atomic absorption spectrometry
- B. Isotope dilution mass spectrometry
- C. Instrumental neutron activation analysis
- D. Flame emission spectrometry
- E. Flameless atomic absorption spectrometry
- F. Ion chromatography
- G. ASTM Methods: (1)D3173, (2)D3174, (3)D3175, (4)D3177, (5)D3178, (6)D3179
- H. Combustion coulometry
- 1. Commercial coal analyzers: moisture, ash, sulfur, Btu, volatile matter, carbon, hydrogen, nitrogen
- J. Commercial calorimeter
- K. Gas chromatography
- L. X-ray fluorescence
- M. Inductively coupled plasma emission spectrometry

#### Analysts

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Page 5 SRM 1632b National Bureau of Standards Ernest Ambler, Director

## National Bureau of Standards

## Certificate of Analysis

#### Standard Reference Material 1633a

#### Trace Elements in Coal Fly Ash

This Standard Reference Material (SRM) is intended for use in the evaluation of analytical methods for the determination of constituent elements in coal fly ash or materials with a similar matrix.

SRM 1633a is a fly ash that was sieved through a No. 170 sieve with a nominal sieve opening of 90 µm.

Certified Values of Constituent Elements: The certified values for the constituent elements are shown in Table 1. The analytical techniques used and the analysts are given in Table 3. The certified values are based on results obtained by reference methods of known accuracy or from two or more independent, reliable analytical methods. Noncertified values are given for information only in Table 2.

Notice and Warnings to Users: This certification is invalid 5 years from date of purchase of the SRM. The constituents certified or analyzed are reviewed periodically and may be updated to reflect improved measurement. Updated certificates will be made available upon request.

<u>Use:</u> This material should be dried to a constant weight before using. Recommended procedures for drying are: (1) Vacuum drying for 24 hours at ambient temperature using a cold trap at or below -50 °C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying for 2 hours in an oven at 105 °C; (3) drying in a dessicator over P<sub>2</sub>O<sub>5</sub> or Mg<sub>2</sub>ClO<sub>4</sub>. Samples of the dried material weighing at least 250-mg should be used for analysis. When not in use the material should be kept in a tightly sealed bottle.

Source and Preparation of Material: The fly ash material was supplied by a coal fired power plant and is a product of Pennsylvania and West Virginia coals. It was selected as a typical fly ash and is not intended as a fly ash from a specific coal or combustion process. The material was sieved and blended for 2 hours in a Vee blender. The material was then removed and placed in a series of bulk containers from which specific samples were taken for homogeneity testing and certification analysis. Twelve bottles were selected for the homogeneity test. Samples from each bottle were analyzed for cobalt, chromium, europium, iron, scandium, and thorium using nondestructive neutron activation analysis. The observed standard deviations for both 50 and 250 mg sample sizes were consistent with counting statistics, indicating that the fly ash is homogeneous within  $\pm$  5% (relative) based on these elements. The homogeneity testing and certification analyses were performed in the NBS Center for Analytical Chemistry.

The overall direction and coordination of the analytical measurements leading to the initial certification were performed in the Center for Analytical Chemistry under the chairmanship of L.A. Machlan.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W.P. Reed and T.E. Gills.

Gaithersburg, MD 20899 January 5, 1985 (Revision of certificate dated April 18, 1979) Stanley D. Rasberry, Chief
Office of Standard Reference Materials

Table 1. Certified Values of Constituent Elements

Major	Content Wt. Percent	Minor	Content
Constituents		Constituents	Wt. Percent
Aluminum Iron Potassium Silicon Calcium	$ 14.3 \pm 1.0^{a}  9.4 \pm 0.1  1.88 \pm 0.06  22.8 \pm 0.8  1.11 \pm 0.01 $	Magnesium Sodium	$0.455 \pm 0.010$ $0.17 \pm 0.01$

#### Trace Constituents

Element	Content µg/g	Element	Content µg/g
Antimony	$6.8 \pm 0.4$	Rubidium	131 ± 2
Arsenic	145 ± 15	Selenium	$10.3 \pm 0.6$
Cadmium	$1.00 \pm 0.15$	Strontium	$830 \pm 30$
Chromium	196 ± 6	Thorium	$24.7 \pm 0.3$
Copper	$118 \pm 3$	Thallium	$5.7 \pm 0.2$
Manganese	179 ± 8	Uranium	$10.2 \pm 0.1$
Mercury	$0.16 \pm 0.01$	Vanadium	$297 \pm 6$
Nickel	127 ± 4	Zinc	$220 \pm 10$
Lead	$72.4 \pm 0.4$		

<sup>&</sup>lt;sup>a</sup>The uncertainties of the certified values are based on judgment and represent an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 250-mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents).

#### Supplemental Information

Note: The following values are not certified because they are not based on the results of either a reference method or of two or more independent methods. These values are included for information only.

Table 2. Noncertified Values for Constituent Elements

	Content		Content
Element	Wt. Percent	Element	<u>μg/g</u>
Barium	0.15	Beryllium	12
Titanium	0.8	Cerium	180
Sulfur	0.18	Cobalt	46
		Cesium	11
		Europium	4
		Gallium	58
		Hafnium	8
		Molybdenum	29
		Scandium	40

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Table 3. Analytical Methods Used for Certified Constituent Elements

Method/ Element	A	В	С	D	E	F	G	Н	I
Aluminum	٠		•						•
Antimony			•				•		
Arsenic	•		•						
Cadmium		•	•	•			•		
Calcium	•	•			•				
Chromium	•	•	•						
Copper	9	•	•						
lron	•	•	•						
Lead		•		•	•				
Magnesium	•	•							
Manganese	٠		•						•
Mercury	٠		•						
Nickel	•	•		•	•				
Potassium	•	•			•				
Rubidium	•	•	•		•				
Selenium	•		•				•		
Silicon					•			•	
Sodium	•		•						
Strontium	•				•	•			
Thallium		•					•		
Thorium		•	•						
Uranium		•							
Vanadium	۰	٠	•						
Zinc	•	•		•	•	•			

#### Analytical Methods

- A. Atomic Absorption Spectrometry or Flame Emission Spectrometry
- B. Isotope Dilution Mass Spectrometry
- C. Neutron Activation Analysis
- D. Polarography
- E. X-ray Fluorescence Spectrometry
- F. Inductively-Coupled Plasma Emission Spectrometry
- G. Isotope Dilution Spark Source Mass Spectrometry
- H. Gravimetry
- 1. Direct Coupled Plasma Emission Spectrometry

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#### Analysts

#### NBS Center for Analytical Chemistry

1.	J. B. Baldwin	16.	W. R. Kelly
2.	T. J. Brady	17.	H. M. Kingston
3.	E. R. Deardorff	18.	E. C. Kuehner
4.	M. G. Dias	19.	R. M. Lindstrom
5.	L. J. Powell	20.	L. A. Machlan
6.	M. S. Epstein	21.	E. J. Maienthal
7.	R. F. Fleming	22.	J. S. Maples
8.	E. L. Garner	23.	J. D. Messman
9.	T. E. Gills	24.	L. J. Moore
10.	C. A. Grabnegger	25.	P. J. Paulsen
11.	J. W. Gramlich	26.	P. A. Pella
12.	R. R. Greenberg	27.	T. C. Rains
13.	S. Hanamura	28.	K. J. R. Rosman
14.	S. H. Harrison	29.	T. A. Rush
15.	E. G. Heald	30.	P. A. Sleeth
		31.	R. L. Watters, Jr

SRM 1633a Page 4 U. S. Department of Commerce Malcolm Baldrige Secretary National Burosu of Standards Ernest Ambler, Director

## National Bureau of Standards Certificate of Analysis

#### Standard Reference Material 1634a

#### Trace Elements in Fuel Oil

This Standard Reference Material is intended for use in the evaluation of methods and the calibration of apparatus used in the analysis of fuel oils and other materials with similar matrices for trace elements. SRM 1634a is a commercial "No. 6" residual fuel oil as defined by the American Society for Testing and Materials (ASTM). This SRM was certified using two or more independent methods of analysis and a single method that has been carefully evaluated with respect to its accuracy and precision. Methods were selected to include those that are commonly used in the field and in laboratories.

The certified values are given in table 1 and are based on at least a 1.0 g sample of the material which is the minimum amount that should be used for analysis.

Table 1						
Element <sup>l</sup>	Content $^2 \mu g/g$	Element <sup>1</sup>	Content <sup>2</sup> , Wt %			
Lead	$2.80 \pm 0.08$					
Manganese <sup>b, c</sup>	$0.19 \pm 0.02$	Sulfur <sup>f,g,h</sup>	$2.85 \pm 0.05$			
Nickel <sup>d, e</sup>	29 ± 1					
Selenium <sup>b, c</sup>	$0.15 \pm 0.02$					
Sodium <sup>b, e</sup>	87 ± 4					
Vanadium <sup>a,b,d</sup>	56 ± 2					
Zinc <sup>b</sup> ,d	$2.7 \pm 0.2$					

- 1. Method of Analysis
  - a. Isotope Dilution Mass Spectrometry
  - b. Neutron Activation Analysis
  - c. Atomic Absorption Spectrometry
  - d. Spark Source Mass Spectrometry
- e. Inductive Coupled Plasma Spectrometry
- f. Gravimetry
- g. Ion Chromatography
- h. X-ray Fluorescence
- The uncertainties shown are expressed at the 95% confidence level and include any observed material heterogeneity, possible method differences, and errors of measurement.

NOTICE: The certification of SRM 1634a is valid for 3 years from date of purchase.

The statistical analysis of the certification data was performed by K.R. Eberhardt of the Statistical Engineering Division.

The overall direction and coordination of the analytical measurements leading to certification were performed in the Inorganic Analytical Research Division, E.L. Garner, Chief.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Washington, D.C. 20234 February 19, 1982 George A. Uriano, Chief Office of Standard Reference Materials

#### PREPARATION, TESTING, AND ANALYSIS

A random scheme for sample selection was used in assessing the homogeneity of this material. The elements calcium and vanadium were measured by x-ray fluorescence as indicators of homogeneity. Based on these elements, the material variability for this lot of 1634a is within  $\pm 2\%$  relative.

Long-term stability of this SRM has not been rigorously established. When not in use, the material should be stored in the tightly sealed bottle. NBS will continue to monitor this material and any substantive change in its certification will be reported to the purchasers.

Analyses for the various elements were performed in the Center for Analytical Chemistry, Inorganic Analytical Research Division, by I. L. Barnes, T.A. Butler, E.R. Deardorff, J.W. Gramlich, S. Hanamura, H.M. Kingston, W.F. Koch, G.M. Lambert, R.M. Lindstrom, L.A. Machlan, J.R. Moody, P.J. Paulson, T.C. Rains, T.A. Rush, and R. Zeisler.

The homogeneity studies were performed in the Gas and Particulate Science Division by P.A. Pella and M. Watson.

The physical properties were measured by S. Weeks, Materials Chemistry Division, Center for Materials Science.

The values and physical properties data in table 2 are not certified because they are based on a non-reference method or were not determined by two or more independent methods. The values are included for information only.

Table 2
Supplemental Information

Element	Content, $\mu g/g$	Physical Properties	
Arsenic	( 0.12 )		
Beryllium	( 0.006 )	Flash Point <sup>a</sup>	64 °C
Bromine	(<1 )		
Cadmium	( 0.002 )	Kinematic Viscosity <sup>b</sup>	321.66
Calcium	(16)	at 50 °C	
Chlorine	(31)	Pour Point <sup>c</sup>	−10 °C
Chromium	( 0.7 )		
Cobalt	( 0.3 )	Density at 20 °C <sup>d</sup>	0.995 g/cm <sup>3</sup>
lron	(31)		
Mercury	(<0.002)		
Molybdenum	( 0.12 )		

#### Methods Used for Physical Tests

- a. ASTM D-93-80 Flash Point by Pensky-Martens Closed Tester
- b. ASTM D445-79 Kinematic Viscosity of Transparent and Opaque Liquids
- c. ASTM D97-66 (1978) Pour Point of Petroleum Oils
- d. ASTM D4052-81 Density and Relative Density of Liquids by Digital Density Meter (modified)

National Bureau of Standards Ernest Ambler, Director

## National Bureau of Standards Certificate of Analysis

#### Standard Reference Material 1635

Trace Elements in Coal (Subbituminous)

This Standard Reference Material is intended for use in the calibration of apparatus and the evaluation of techniques employed in the trace element analysis of coal and similar materials. The material should be dried without heat to constant weight before use.

The recommended procedures for drying are either vacuum drying at ambient temperature for 24 hours, or freeze drying in which the drying chamber is kept at room temperature. The moisture content of this material is approximately 20%. Because of this moisture level, it is recommended that small individual samples be dried immediately before use. Drying of large samples may result in a violent discharge of water vapor and resultant loss of sample. When not in use, the material should be kept in a tightly sealed bottle and stored in a cool, dark place. Long-term (>1 year) stability of this SRM has not been rigorously established. NBS will continue to monitor this material and any substantive change will be reported to purchasers.

The certified values given below are based on at least a 250-mg sample of the dried material, the minimum amount that should be used for analysis.

Element 1	Content, $\mu g/g^2$	Element 1	Content, $\mu g/g^2$
Arsenic <sup>a,b</sup>	$0.42 \pm 0.15$	Thorium <sup>c, e</sup>	$0.62 \pm 0.04$
Cadmium <sup>c,d,e</sup>	$0.03 \pm 0.01$	Urani <b>um</b> <sup>c</sup>	$0.24 \pm 0.02$
Chromium <sup>c, e</sup>	$2.5 \pm 0.3$	Vanadium <sup>e, g</sup>	$5.2 \pm 0.5$
Copper <sup>a,c,e</sup>	$3.6 \pm 0.3$	Zinc <sup>c, d</sup>	$4.7 \pm 0.5$
Lead <sup>c,d</sup>	$1.9 \pm 0.2$		
Manganese <sup>a, e</sup>	21.4 ± 1.5	Element <sup>l</sup>	Wt. % <sup>2</sup>
Nickel <sup>c,d</sup>	$1.74 \pm 0.10$	Iron <sup>c, d, e, f</sup>	$0.239 \pm 0.005$
Selenium <sup>a, e</sup>	$0.9 \pm 0.3$	Sulfur <sup>f,h</sup>	$0.33 \pm 0.03$

- I. Methods of Analysis:
  - a. Atomic Absorption Spectrometry
  - b. Photon Activation
  - c. Isotope Dilution Mass Spectrometry
- d. Polarography

- e. Neutron Activation
- f. Spectrophotometry
- g. Flame Emission Spectrometry
- h. Gravimetry
- 2. The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 250-mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.)

The overall direction and coordination of the analytical measurements leading to this certificate were performed in the Analytical Chemistry Division under the chairmanship of L. J. Moore.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by W. P. Reed.

Washington, D.C. 20234 August 22, 1979 (Revision of Certificate dated 1-23-78)

(over)

George A. Uriano
Office of Standard Reference Materials

#### PREPARATION, TESTING, and ANALYSIS

This material was prepared from one lot of subbituminous coal from the Eagle Mine of The Imperial Coal Company, Erie, Colorado. The material was ground and sieved thru a No. 65 (230  $\mu$ m) sieve by the Colorado School of Mines Research Institute. The material was then blended in a V-type blender.

Samples for homogeneity testing were taken from the top, middle, and bottom of three bulk containers of coal, and analyzed by neutron activation analysis for sodium, scandium, chromium, iron, cobalt, lanthanum, cerium, and thorium. Replicate analyses of 250-mg samples indicated a homogeneity for these elements of  $\pm 2.5\%$  (relative) except for chromium, which was homogeneous within counting statistics of  $\pm 6\%$ . The homogeneity measurements were performed in the NBS Analytical Chemistry Division by R. R. Greenberg. Certification analyses for the various elements were made in the NBS Analytical Chemistry Division by T. J. Brady, B. I. Diamondstone, L. P. Dunstan, M. S. Epstein, M. Gallorini, E. L. Garner, T. E. Gills, J. W. Gramlich, R. R. Greenberg, S. H. Harrison, G. M. Hyde, G. J. Lutz, L. A. Machlan, E. J. Maienthal, J. D. Messman, T. J. Murphy, and T. C. Rains.

The following values are *not certified* because they were based on a non-reference method, o were not determined by two or more independent methods. They are included for information only.

Element	Content (μg/g)
Antimony	(0.14)
Cerium	(3.6)
Cobalt	(0.65)
Europium	(0.06)
Gallium	(1.05)
Hafnium	(0.29)
Scandium	(0.63)
	(wt. %)
Aluminum	(0.32)
Sodium	(0.24)
Titanium	(0.02)

National Bureau of Standards Ernest Ambler, Director

## National Bureau of Standards Certificate of Analysis

#### Standard Reference Materials 1636a, 1637a, 1638a

#### Lead in Reference Fuel

This Standard Reference Material is intended for use in the calibration of instruments and the evaluation of techniques used for the analysis of lead in gasoline. Samples of the reference fuel are supplied at four lead concentrations, nominally 0.03, 0.05, 0.07, and 2.0 g/gal. These Standard Reference Materials are made up of various combinations of the four concentrations, see Table 1 on the back of this certificate.

Certified Values: The certified values for the lead content, expressed in units of  $\mu g/g$ , are shown below. These certified values are based on results obtained by isotope dilution mass spectrometry, a definitive method of known accuracy.

	Nominal	Certified
Vial	Lead Concentration	Lead Concentration
Identification	g/gal	μg/g
I	0.03	$11.2\pm0.2^{a}$
II	0.05	$18.8 \pm 0.1$
III	0.07	$25.1 \pm 0.2$
1V	2.0	764 ± 4

<sup>&</sup>lt;sup>a</sup>The uncertainties shown are the 95 percent confidence intervals for a single determination plus allowance for known sources of possible error.

Use: The certification of these materials is based on a minimum sample size of 1.0 gram and only samples equal to or greater than 1 gram should be used for any analytical determination to be related to the certified values of this certificate.

Stability: The ampoules should be stored at temperatures between 10-30 °C. They should not be exposed to intense sources of radiation, including ultraviolet lamps or sunlight. The ampoules should be opened only at time of use. No attempt should be made to keep the material in opened ampoules for future use.

Source and Preparation of Material: The reference fuel containing lead at the four concentration levels were supplied by Phillips Petroleum Company of Bartlesville, Oklahoma. The 91-octane number (Research Octane Number) reference fuel is a mixture of 91 percent by volume (0.899 mole-fraction) 2,2,4,-trimethylpetane and 9 percent by volume (0.101 mole-fraction) n-heptane. Lead was added in the form of tetraethyl lead motor mix.

Analyses leading to certification were performed in the Inorganic Analytical Research Division by T. J. Murphy and I. L. Barnes.

The overall direction and coordination of the technical measurements leading to this certificate were performed by E. L. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of these Standard Reference Materials were coordinated through the Office of Standard Reference Materials by T. E. Gills.

Washington, D.C. 20234 February 5, 1980

George A. Uriano, Chief
Office of Standard Reference Materials

Table 1 Composition of SRM's 1636a, 1637a, 1638a

SRM	Nominal Concentration	No. Units.
1636a	0.03,0.05,0.07, 2.0 g/gal	3 vials each
1637a	0.03,0.05,0.07 g/gal	4 vials each
1638a	2.0 g/gal	12 vials each

Additional Information: Because the volume of the reference material varies with temperature, the various concentrations of lead are certified by weight, i.e., micrograms of lead per gram of fuel. For convenience to the user, information is given for the concentration in the customary units, grams per gallon and grams per liter, at 23 °C. These data are shown in Table 2.

Table 2

Vial	Nominal Concentration	Density <sup>a</sup> at 23 °C	Lead Concentration at 23 °C	
Identification	g/gal	g/mL	g/gal	g/L
I	0.03	0.6888	0.0292	0.0077
II	0.05	0.6888	0.0490	0.0129
III	0.07	0.6888	0.0654	0.0173
IV	2.0	0.6895	1.994	0.527

<sup>&</sup>lt;sup>a</sup>The density (ρ) of each concentration was measured at 23 °C using a modification of ASTM Method D1217. The temperature coefficient of these materials is 0.0008 g.(mL)<sup>-1</sup>.(°C)<sup>-1</sup>

Eq. 1 
$$C_{g/gal} = \frac{3.785 \rho C_{\mu g/g}}{10^3}$$
  
Eq. 2  $C_{g/L} = \frac{\rho C_{\mu g/g}}{10^3}$ 

<sup>&</sup>lt;sup>b</sup>The conversion of the certified values ( $\mu g/g$ ) to C(g/gal) and C(g/L) was done using equations 1 and 2 respectively.

U. S. Department of Commerce
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Ernest Ambler, Director

## National Bureau of Standards Certificate of Analysis

### Standard Reference Material 1641b

Mercury in Water - µg/mL

This Standard Reference Material is intended for use in the primary calibration of instruments and techniques used for the determination of mercury in natural waters. It is designed for the preparation of calibration solutions and for use as a "spike" sample in a "method-of-additions" type analytical procedure.

Mercury concentration

 $1.52 \pm 0.04 \ \mu g/mL$ 

The estimated uncertainty,  $0.04 \,\mu g/mL$ , includes the effects of the observed random variability and an upper bound estimate of possible systematic errors. The random variability expressed as two standard deviations of the certified value is  $\pm 0.02 \,\mu g/mL$  and reflects both internal and between-method variabilities for the NBS atomic absorption and neutron activation measurements. The upper bound estimate of possible systematic errors is  $\pm 0.02 \,\mu g/mL$ .

Stability: The long-term stability of trace mercury solutions has been a constant problem. At or below the  $\mu g/mL$  level, mineral acid stabilization is not sufficient. However, the addition of trace gold to a nitric acid solution of mercury was found to stabilize the concentration of mercury in the two previous issues of this Mercury in Water SRM. Although the mercury concentration of SRM 1641b has not changed significantly in eight months, the stability will continue to be monitored. However, SRM 1641b should not be used after ONE YEAR FROM date of purchase.

<u>Precautions</u>: Traces of mercury vapor are present in most laboratory situations. Therefore, contamination of reagents, equipment, and common laboratory materials may cause a severe problem. Apparatus for analysis at this level must be scrupulously cleaned immediately before use, and only the purest-grade reagents, with respect to mercury, should be used.

SRM 1641b was prepared by J.R. Moody. Atomic absorption analyses were performed by T.C. Rains and T.A. Butler; and neutron activation analyses by R. Zeisler, Inorganic Analytical Research Division.

The overall direction and coordination of technical measurements leading to certification were performed under the chairmanship of E.L. Garner, Inorganic Analytical Research Division. The statistical evaluation was done by R.C. Paule

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Analytical: Two independent techniques were used in the certification of this Standard Reference Material: atomic absorption spectrometry and instrumental neutron activation analysis.

Use: This SRM consists of six ampoules, each containing approximately 20 mL of solution. Dilutions may be made by the addition of accurately measured aliquots, withdrawn from an ampoule, to known volumes of pure or natural water (spiking mode) using conventional techniques. Blank determinations should be made of the water and other reagents used.

The reliability of the dilution process will depend on the care exercised and the reliability of the calibration of the volumetric apparatus, which should have an uncertainty no greater than one percent. The volumetric apparatus should be scrupulosuly cleaned. Diluted solutions should be used without delay, as their stability cannot be guaranteed. SRM 1642b, which is certified for mercury at the ng/mL level, should be used to validate methodology for these concentrations. The long-term retention of unused portions of this Standard Reference Material in opened ampoules is not recommended.

Washington, D.C. 20234 April 13, 1903 George A. Uriano, Chief Office of Standard Reference Materials U. S. Department of Commerce Malcolm Buldrige Secretary National Bureau of Standards Ernest Ambler, Director

## National Bureau of Standards

### Certificate

#### Standard Reference Material 1642b

Mercury in Water - ng/mL

This Standard Reference Material is intended for use in the primary standardization of instruments and techniques used for the determination of mercury in water. It is intended for use as received, without dilution or other alteration. The concentration of mercury in this Standard Reference Material is at, or near, the detection limit of most commercial instruments used for the determination of mercury in water. It is to be used for the primary standardization of these instruments near these detection limits where many analytical problems occur.

Mercury Concentration 1.49 ± 0.06 ng/mL

The estimated uncertainty, 0.06, includes the effects of the observed random variability and an upper bound estimate of possible systematic errors. The random variability expressed as two standard deviations of the certified value is  $\pm 0.04$  and reflects both internal and between-method variabilities for the NBS atomic absorption and neutron activation measurements. The upper bound estimate of possible systematic errors is  $\pm 0.02$ .

Stability: Trace mercury solutions have been a constant problem when long-term storage is required. Below the  $\mu$ g, mL level, mineral acid stabilization is not sufficient. A stabilizing technique has been applied to this Standard Reference Material that allows for prolonged storage. Gold, as the tetrachloride, has been added in a concentration 10 times that of the mercury. The gold ion, in conjuction with the normal mineral acid, has proven to be an effective stabilizer. It is recommended that this Standard Reference Material not be used after ONE YEAR FROM DATE OF PURCHASE.

<u>Precautions:</u> Traces of mercury vapor are present in most laboratory situations. Therefore, contamination of reagents, equipment, and common laboratory materials is a severe problem. Apparatus for analyses at this level must be scrupulously cleaned immediately before use, and only the purest-grade reagents should be employed. After use, the bottle should be capped tightly and placed inside the aluminized bag, which should be folded and sealed with a sealing tape. This safeguard will assist in maintaining the integrity of the sample.

<u>Analytical:</u> Two independent techniques were used in the certification of this Standard Reference Material: atomic absorption spectroscopy and neutron activation analysis.

<u>Usc:</u> This Standard Reference Material should be used, as received, without dilution. It may be carried through the chemical manipulations required for the analytical procedure normally used for the analysis of natural waters.

This Standard Reference Material was prepared by J.R. Moody. Atomic absorption analyses were performed by 1.C. Rains and T.A. Butler and neutron-activation analyses were performed by R. Zeisler.

The overall direction and coordination of the technical measurements leading to the certification were performed under the chairmanship of E.L. Garner. The statistical evaluation was done by R.C. Paule.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234 July 15, 1982 George A. Uriano, Chief Office of Standard Reference Materials U. S. Department of Commerce
Malcolm Baldrige
Secretary
National June to O Standards
Ernest Ambler, Director

### National Bureau of Standards

### Certificate

#### Standard Reference Material 1643b

#### Trace Elements in Water

This Standard Reference Material (SRM) is intended primarily for use in evaluating the accuracy of trace element determinations in filtered and acidified fresh water and for calibrating instrumentation used in these determinations. SRM 1643b consists of approximately 950 mL of water in a polyethylene bottle, which is sealed in an aluminized bag to maintain stability. SRM 1643b simulates the elemental composition of fresh water. Nitric acid is present at a concentration of 0.5 moles per liter to stabilize the trace elements.

Concentrations of Constituent Elements: The concentrations of the trace elements that were determined are shown in Table 1. The certified values are based on results obtained either by reference methods of known accuracy or by two or more independent, reliable analytical methods. Noncertified values, which are given for information only, appear in parentheses.

#### Notice and Warnings to Users:

Expiration of Certification: This certification is invalid two years after the shipping date.

<u>Precautions:</u> The bottle should be shaken before use because of possible water vapor condensation. To prevent possible contamination of the SRM, do not insert pipets into the bottle. After use, the bottle should be capped tightly and placed inside the aluminized bag, which should be folded and sealed with sealing tape. This safeguard will protect the SRM from possible environmental contamination and long-term loss of water.

Elemental determinations of ng/g levels are limited by contamination. Apparatus should be scrupulously cleaned and only the purest grade reagents employed. Sampling and manipulations, such as evaporations, should be done in a clean environment, for example, a Class 100 clean hood.

The overall direction and coordination of the technical measurements leading to this certification were performed under the direction of E. Garner, Chief of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, DC 20234 May 18, 1984 Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(Table 1)
Concentrations of Constituent Elements

	Concentration,*		Concentration,*
Element	ng/g	Element	ng/g
Arsenic <sup>1,5</sup>	(49)**	Lead <sup>3,4b</sup>	$23.7 \pm 0.7$
Barium <sup>2a,2b,5</sup>	44 ± 2	Manganese <sup>1, 2a, 3</sup>	$28 \pm 2$
Beryllium <sup>1,2a</sup>	19 ± 2	Molybdenum <sup>2a, 5</sup>	85 $\pm 3$
Bismuth 1	(11)	Nickel <sup>2a, 3</sup>	49 ± 3
Boron <sup>2a</sup>	(94)	Selenium <sup>1,5</sup>	$9.7 \pm 0.5$
Cadmium <sup>2b,3,5</sup>	20 ± 1	Silver <sup>1,5</sup>	$9.8 \pm 0.8$
Chromium <sup>4b</sup>	$18.6 \pm 0.4$	Strontium <sup>2a, 5</sup>	227 $\pm 6$
Cobalt <sup>1,5</sup>	26 ± 1	Thallium <sup>4b</sup>	$8.0 \pm 0.2$
Copper <sup>3,4b</sup>	$21.9 \pm 0.4$	Vanadium <sup>4b</sup>	$45.2 \pm 0.4$
Iron <sup>2a, 4a, 5</sup>	99 ± 8	Zinc <sup>2a,5</sup>	66 ± 2

The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision and possible systematic errors among methods. To convert to nanograms per milliliter, multiply by the density of the SRM. The density at 23 °C is 1.017 grams per milliliter.

\*\* Values in parentheses are not certified.

- 1. Atomic absorption spectrometry, electrothermal
- 2. Atomic emission spectrometry,
- a. de plasma
- b. flame
- 3. Laser enhanced ionization flame spectrometry
- 4. Isotopic dilution mass spectrometry,
  - a. resonance ionization
  - b. thermal ionization
  - Neutron activation, instrumental

Source and Preparation of Material: SRM 1643b was prepared at the U.S. Geological Survey, National Water Quality Laboratory, Arvada, Colorado, under the direction of V.J. Janzer of that laboratory and J.R. Moody of the NBS Center for Analytical Chemistry. Only high-purity reagents were used and the containers were acid-cleaned and sterilized before use. In the preparation, a polyethylene cylindrical tank was filled with distilled water and sufficient nitric acid to make the solution approximately 0.5 moles HNO<sub>3</sub> per liter. Solutions containing known amounts of calcium, sodium, magnesium, potassium, and the elements to be determined were added to the acidified water solution with constant stirring. After thoroughly mixing, the solution was filtered, sterilized, and then transferred to one-liter polyethylene bottles. The approximate concentrations, in  $\mu g/mL$ , of Ca, Na, Mg, and K are respectively 35, 8, 15, and 3.

#### Analysts:

Center for Analytical Chemistry, National Bureau of Standards

1. K. A. Brletic	10. J. R. Moody
2. T. A. Butler	11. L. J. Powell
3. E. C. Deal	12. T. C. Rains
4. M. S. Epstein	13. T. A. Rush
5. J. D. Fassett	14. S. F. Stone
6. K. Fitzpatrick	15. G. C. Turk
7. H. M. Kingston	16. R. L. Watters, Jr.
8. R. M. Lindstrom	17. R. Zeisler

U. S. Department of Commerce
Malcolm-Baldrige
Secretary
Secretary
National Bureau of Standards
Ernest Ambler, Director

## National Bureau of Standards Certificate

# Standard Reference Material 1644 Generator Columns for Polynuclear Aromatic Hydrocarbons

SRM 1644 is intended to provide accurate concentrations of anthracene, benzo(a) anthracene (1,2-benzanthracene), and benzo(a)pyrene (3,4-benzpyrene) in water. The SRM consists of three 50 cm x 0.6 cm (coiled) stainless steel tubes, each packed with fine quintus quartz (sea sand) coated with approximately 0.5 percent by weight of the polynuclear aromatic hydrocarbon (PAH) of interest.

Principle of Operation: A saturated aqueous solution of the PAH of interest is generated by flowing high-purity water slowly through the column. Because the aqueous solubility of a compound is a well-defined thermodynamic quantity, a saturated solution has a fixed concentration (1, 2, 3).

Equilibration and Use of Generator Columns: To equilibrate a new column before initial use, purge with high-purity water, such as commercial HPLC grade water. The volume required for equilibration of each column is: 500 mL for anthracene, 1000 mL for benzo(a)anthracene, and 500 mL for benzo(a)pyrene. After equilibration, pump the high-purity water at a constant temperature (±0.1 °C) through the column at a flow rate between 0.1 and 5 mL/min to produce a saturated solution. Record the temperature. The solution should be used immediately after generation to avoid sorption losses.

If either the temperature is changed by as much as 1 °C or the flow is interrupted for less than one hour, pump 25 mL water through the column under the new conditions to restore equilibrium prior to sample collection. However, if the flow is interrupted for more than one hour, pump 50 mL water prior to sample collection. During periods of frequent use, a column can be kept equilibrated by maintaining a steady, but low, flow rate of approximately 0.1 mL/min through the column. The flow rate can be increased to collect a large volume of sample and then decreased again. Columns should be purged with 10 liters of oil-free nitrogen prior to storage periods of more than one month.

Certified Concentrations: When used as directed, these columns generate saturated solutions. The concentrations of the compounds in these solutions at temperatures between 10 and 30 °C were determined by two independent analytical methods. The data obtained were combined by fitting an empirical expression of the form  $\ln[\text{Conc}] = A + B(1/T) + C(1/T^2)$  by least squares. In this equation,  $\ln[\text{Conc}]$  is the natural logarithm of the concentration, T is the absolute temperature, and A, B, and C are constants for each compound. The derived equations for anthracene, benzo(a)anthracene, and benzo(a)pyrene were used to calculate the certified concentrations at one degree intervals between 10 and 30 °C. These certified concentrations are given in Tables I-III.

Service Life of Columns: Generator columns for anthracene, benzo(a)anthracene, and benzo(a)pyrene are certified for either two years or for total aqueous purge volumes of 7.5 x 10<sup>2</sup>, 3 x 10<sup>3</sup>, and 1.5 x 10<sup>4</sup> liters, respectively, whichever comes first.

Consultation on the statistical design of the experimental work and statistical analysis of the data was provided by K. R. Eberhardt of the Statistical Engineering Division. Coordination of the technical measurements leading to certification was performed by W. E. May, R. A. Velapoldi, and H. S. Hertz. Technical measurements leading to the development and certification of SRM 1644 were performed by the following members of the Center for Analytical Chemistry: W. E. May, J. M. Brown-Thomas, W. J. Sonnefeld, R. A. Velapoldi, and P. A. White.

The technical and support aspects concerning the certification and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234 April 27, 1981 George A. Uriano, Chief Office of Standard Reference Materials

The anthracene generator column, after equilibration, produces aqueous solutions that contain very small amounts of phenanthrene. Because of its relatively high solubility ( $1000 \,\mu\text{g/kg}$ ), residual amounts of phenanthrene are depleted from the column at a rapid rate.

The benzo(a)anthracene generator column, after equilibration, produces aqueous solutions that contain as much as  $0.3 \mu g/kg$  anthracene in addition to the certified concentrations of benzo(a)anthracene. The concentration of anthracene in the generator column effluent varies with time (volume) and, therefore, is not certified.

The benzo(a)pyrene generator column, after equilibration, produces aqueous solutions that contain as much as  $0.1 \mu g/kg$  benzo(a)anthracene and  $1 \mu g/kg$  chrysene in addition to the certified concentrations of benzo(a)pyrene. The concentration of these non-analyte components vary with time and, therefore, are not certified.

Analyses of the Saturated Aqueous Solutions: The concentrations of the saturated aqueous solutions of anthracene, benzo(a)anthracene, and benzo(a)pyrene in the effluent from the respective generator columns were determined by two independent analytical techniques. The first technique was high-performance liquid chromatography (HPLC). It involved quantitative extraction of the PAH of interest from the aqueous effluent by an "extractor column" packed with an octadecylsilane (C<sub>18</sub>) bonded phase; use of an acetonitrile-water eluant to transfer components from the extractor column to an analytical C<sub>18</sub> column for separation of the analyte from non-analyte components; and detection of the analyte by measuring its absorbance at 254 nm. The second technique involved the use of a "standard addition" spectrofluorimetric technique for "on stream" analysis. The aqueous effluent from the generator column was mixed with PAH standards dissolved in acetonitrile and the PAH concentration of the resultant mixture was determined by fluorescence at the following excitation ( $\lambda_{ex}$ ) and emission ( $\lambda_{em}$ ) wavelengths: anthracene,  $\lambda_{ex} = 254$  nm,  $\lambda_{em} = 384$  and 404 nm; benzo(a)anthracene,  $\lambda_{ex} = 290$  nm,  $\lambda_{em} = 395$  nm; benzo(a)pyrene,  $\lambda_{ex} = 296$  nm,  $\lambda_{em} = 414$  nm. The PAH effluent concentration was determined mathematically. Where necessary, corrections for inner filter effects or the emission-absorbance contributions by impurities were made to obtain the values for determining the certified PAH concentrations listed in Tables I-III.

#### References:

- 1. May, W. E., The Solubility Behavior of Polycyclic Armomatic Hydrocarbons in Aqueous Systems, American Chemical Society Advances in Chemistry Series 185 (7), 143-192 (1980).
- 2. May, W. E., Wasik, S. P., and Freeman, D. H., Determination of the Aqueous Solubility of Polynuclear Aromatic Hydrocarbons by a Coupled-Column Liquid Chromatographic Technique, Anal. Chem. <u>50</u>, 1 (1978).
- 3. Schwarz, F. P. and Miller, J. M., Determination of the Aqueous Solubilities of Organic Liquids at 10°C, 20°C, and 30°C by Elution Chromatography, Anal. Chem. <u>52</u>, 2162-2164 (1980).

Table I. Certified Aqueous Concentrations of Anthracene and Their Uncertainties, in Micrograms/Kilogram and Nanomoles/Liter, as a Function of Temperature

Temperature °C	Concentration and I $\mu g/kg$	ts Uncertainty <sup>1</sup> , nmol/L
10	$16.6 \pm 0.7$	93.1 ± 4.0
11	$17.6 \pm 0.6$	98.7 ± 3.6
12	$18.7 \pm 0.6$	$105 \pm 3.3$
13	$19.8 \pm 0.5$	$111 \pm 3.1$
14	$21.1 \pm 0.5$	118 ± 2.9
15	$22.4 \pm 0.5$	126 ± 2.9
16	$23.8 \pm 0.5$	134 ± 2.9
17	$25.4 \pm 0.5$	142 ± 2.9
18	$27.0 \pm 0.5$	151 ± 3.0
19	$28.8 \pm 0.5$	161 ± 3.1
20	$30.7 \pm 0.6$	$172 \pm 3.2$
21	$32.8 \pm 0.6$	$184 \pm 3.2$
22	$35.0 \pm 0.6$	196 ± 3.2
23	$37.4 \pm 0.6$	$210 \pm 3.2$
24	$39.9 \pm 0.6$	$224 \pm 3.3$
25	$42.7 \pm 0.6$	$239 \pm 3.7$
26	$45.7 \pm 0.8$	$256 \pm 4.4$
27	$48.9 \pm 1.0$	273 ± 5.5
28	$52.4 \pm 1.3$	293 ± 7.2
29	$56.1 \pm 1.7$	$313 \pm 9.5$
30	$60.1 \pm 2.2$	$336 \pm 12$

<sup>&</sup>lt;sup>1</sup>The uncertainties are 99 percent (Working-Hotelling) confidence bands for the entire regression curve. The difference between the true and certified concentrations should be less than the stated uncertainty at the 99 percent confidence level.

Table II. Certified Aqueous Concentrations of Benzo(a)anthracene and Their Uncertainties, in Micrograms/Kilogram and Nanomoles/Liter, as a Function of Temperature

Temperature	Concentration and	Its Uncertainty <sup>1</sup> ,
°C	μg/kg	nmol/L
10	$3.38 \pm 1.2$	$14.8 \pm 5.3$
11	$3.60 \pm 1.1$	$15.8 \pm 4.6$
12	$3.83 \pm 0.91$	$16.8 \pm 4.0$
13	$4.09 \pm 0.79$	$17.9 \pm 3.4$
14	$4.36 \pm 0.68$	$19.1 \pm 3.0$
15	$4.65 \pm 0.59$	$20.4 \pm 2.6$
16	$4.96 \pm 0.54$	$21.7 \pm 2.4$
17	$5.29 \pm 0.55$	$23.2 \pm 2.4$
18	$5.65 \pm 0.60$	$24.8 \pm 2.6$
19	$6.04 \pm 0.68$	$26.4 \pm 3.0$
20	$6.45 \pm 0.77$	$28.2 \pm 3.4$
21	$6.90 \pm 0.87$	$30.2 \pm 3.8$
22	$7.38 \pm 0.94$	$32.3 \pm 4.1$
23	$7.90 \pm 1.0$	$34.5 \pm 4.4$
24	$8.45 \pm 1.0$	$36.9 \pm 4.5$
25	$9.05 \pm 1.0$	$39.5 \pm 4.6$
26	$9.69 \pm 1.0$	$42.3 \pm 4.6$
27	$10.4 \pm 1.1$	$45.4 \pm 4.7$
28	$11.1 \pm 1.2$	$48.4 \pm 5.0$
29	$11.9 \pm 1.3$	51.9 ± 5.9
30	$12.8 \pm 1.7$	$55.8 \pm 7.3$

The uncertainties are 99 percent (Working-Hotelling) confidence bands for the entire regression curve. The difference between the true and certified concentrations should be less than the stated uncertainty at the 99 percent confidence level.

Table III. Certified Aqueous Concentrations of Benzo(a)pyrene and Their Uncertainties, in Micrograms/Kilogram and Nanomoles/Liter, as a Function of Temperature

Temperature °C	Concentration and µg/kg	Its Uncertainty <sup>1</sup> , nmol/L
10	$0.59 \pm 0.06$	$2.34 \pm 0.23$
11	$0.63 \pm 0.05$	$2.50 \pm 0.20$
12	$0.67 \pm 0.04$	$2.65 \pm 0.17$
13	$0.71 \pm 0.04$	$2.81 \pm 0.15$
14	$0.76 \pm 0.03$	$3.01 \pm 0.12$
15	$0.81 \pm 0.03$	$3.21 \pm 0.11$
16	$0.87 \pm 0.03$	$3.44 \pm 0.10$
17	$0.93 \pm 0.03$	$3.68 \pm 0.10$
18	$0.99 \pm 0.03$	$3.92 \pm 0.11$
19	$1.06 \pm 0.03$	$4.19 \pm 0.12$
20	$1.13 \pm 0.03$	$4.47 \pm 0.13$
21	$1.21 \pm 0.04$	$4.79 \pm 0.15$
22	$1.30 \pm 0.04$	$5.14 \pm 0.16$
23	$1.39 \pm 0.04$	$5.50 \pm 0.16$
24	$1.49 \pm 0.04$	$5.89 \pm 0.17$
25	$1.59 \pm 0.04$	$6.28 \pm 0.17$
26	$1.71 \pm 0.04$	$6.76 \pm 0.18$
27	$1.83 \pm 0.05$	$7.23 \pm 0.19$
28	$1.96 \pm 0.05$	$7.74 \pm 0.22$
29	$2.11 \pm 0.07$	$8.33 \pm 0.27$
30	$2.26 \pm 0.09$	$8.92 \pm 0.35$

<sup>&</sup>lt;sup>1</sup>The uncertainties are 99 percent (Working-Hotelling) confidence bands for the entire regression curve. The difference between the true and certified concentrations should be less than the stated uncertainty at the 99 percent confidence level.

U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis Standard Reference Material 1645

#### River Sediment

This Standard Reference Material (SRM) is intended for use for the calibration of apparatus and the vermeation of methods used in the analysis of river sediments and material with a similar matrix.

Certified Values of Constituent Elements: The certified values for the constituent elements are shown in Table 1. The analytical techniques used and the names and affiliations of the analysts are shown in Table 3. Certified values are based on results obtained by reference methods of known accuracy and analyses performed by two or more analysts; or alternatively, from results obtained by two or more independent, reliable analytical methods. Noncertified values are given for information only in Table 2. All values are based on measurements made on a dried sample of at least 100 mg for all constituents except iron and chromium for which a 1-g sample was used.

Notice and Warnings to Users:

Expiration of Certification: This certification is invalid 5 years from the date of purchase.

Stability: This material has been freeze-dried and is essentially free of moisture. However, its stability has not been rigorously assessed. NBS will continue to monitor this material and if substantive changes in certification occur the purchasers will be notified. The material should be kept in its original bottle and stored at temperatures between 10-30 °C. The material should be dried without heat to a constant weight before using. Recommended procedures for drying are: (1) drying for 24 hours using a cold trap at or below -50 °C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying for 24 hours in a desiccator over P<sub>2</sub>O<sub>5</sub> or Mg (ClO<sub>4</sub>)<sub>2</sub>.

Use: Material of this kind is intrinsically heterogeneous. Consequently, the analyst should endeavor to minimize any segregation by thoroughly mixing the contents of the bottle by shaking and/or rolling before each use. In addition, when taking a portion for analysis, the analyst should strive to remove as representative a sample as possible.

Source and Preparation of Material: The material for this SRM was prepared from material dredged from the bottom of the Indiana Harbor Canal near Gary, Indiana. The material was screened to remove foreign objects, freeze-dried, and sieved to pass a No. 80 (180 µm) screen. The material was thoroughly mixed in a V-blender and bottled. The bulk material was radiation-sterilized to minimize alteration due to biological activity.

The collection, freeze-drying and homogenization of this SRM were performed under the supervision and direction of H.L. Rook, Gas and Particulate Science Division.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J.K. Taylor, Center for Analytical Chemistry.

The technical and support aspects involved in the preparation, current and previous certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills and W.P. Reed.

Washington, D.C. 20234 May 5, 1982 (Revision of Certificate dated 11-16-78) George A. Uriano, Chief Office of Standard Reference Materials

Homogeneity Assessment and Certification: The homogeneity of this material was established using a minimum sample size of 100 milligrams for all constituents except iron and chromium for which the sample size was 1.0 gram.

Randomly selected bottles were used for the analytical measurements. Each analyst examined at least 6 different bottles, some of them measuring replicate samples from each bottle. Accordingly, it is believed that all bottles of this SRM have substantially the same composition. Measurements and calibrations were made to reduce random and systematic errors to no more than one percent, relative.

Table 1. Certified Values of Constituent Elements

Major Constituents		Minor Constituents	
Element	Content wt. percent a	Element	Content wt. percent <sup>a</sup>
Aluminumb	$2.26 \pm 0.04$	Magnesium <sup>b</sup>	$0.74 \pm 0.02$
Chromium	$2.96 \pm 0.28$	Sodium <sup>b</sup>	$0.54 \pm 0.01$
lron	$11.3 \pm 1.2$	Zinc	$0.172 \pm 0.017$
Potassium <sup>b</sup>	$1.26 \pm 0.05$		

#### Trace Constituents

	Content		Content
Element	$\mu g/g^a$	Element	$\mu g/g^a$
Cadmium	$10.2 \pm 1.5$	Nickel	45.8 ± 2.9
Copper Cobalt <sup>b</sup>	109 ± 19	Thallium	$1.44 \pm 0.07$
Cobalt <sup>b</sup>	$10.1 \pm 0.6$	Thorium	$1.62 \pm 0.22$
Lead	$714 \pm 28$	Uranium	$1.11 \pm 0.05$
Manganese	785 $\pm$ 97	Vanadium	$23.5 \pm 6.9$
Mercury	$1.1 \pm 0.5$		

<sup>&</sup>quot;The uncertainties of the certified values for the elements, except those noted by superscript "b," include those errors associated with both measurement and material variability. They represent the 95 percent tolerance limits for an individual sub-sample, i.e., 95 percent of the sub-samples from a unit of this SRM would be expected to have a composition within the indicated range of values 95 percent of the time.

<sup>&</sup>lt;sup>h</sup>These elements are certified as a part of the NBS update certification program. For each element a "best value" is given based on all methods of measurement that were used as well as a *standard error* of this value. Both are based on considerations of variability both within and between analytical methods.

#### Supplemental Information

Note: The following values are not certified because they are not based on the results of either a reference method or of two or more independent methods. These values are included for information only.

Table 2. Noncertified Values for Constituent Elements

	Content		Content
Element	wt. Percent	Element	μg, g
Calcium	(2.9)	Antimony	(51)
Fluorine	(0.09)	Arsenic	(66)
Sulfur	(1.1)	Lanthanum	(9)
		Scandium	(2)
		Selenium	(1.5)

Additional Information: The values listed below are based on measurements made in one laboratory and while no reason exists to suspect systematic bias in these numbers, no attempt was made to evaluate such bias attributable to either the method or the laboratory. The method used for each set of measurements is also listed. The indicated uncertainties are two times the standard deviation of the mean. These values are included for information only.

Table 3

	Content
Constituent	wt. percent
Kjeldahl Nitrogen	$(0.0797\% \pm 0.0048)$
Total Phosphorus	$(0.051\% \pm 0.001)[1]$
Loss on Ignition (800 °C)	$(10.72\% \pm 0.28)$
Oil and Grease (Freon)	$(1.71\% \pm 0.26)[3]$
Chemical Oxygen Demand (Dichromate)	$(149,400 \text{ mg/kg} \pm 9,000)[2]$

#### References

- 1. ASTM Method E-350
- 2. Standards Methods for the Examination of Water and Waste Water, 14th Edition (1975), Section 508, pp 550.
- 3. Ibid., Section 502, pp 518.

Table 3A Methods and Analysts

Method/					1	
Element	A	В	С	D	E	F
Aluminum	•		•		•	
Arsenic			•			
Antimony	•	1				
Cadmium			•	•		
Calcium			•			
Chromium			•			
Cobalt			•		•	
Copper		•	•			
Fluorine						•
Iron	۰		•			
Lanthanum			•			
Lead		•		•		
Magnesium	•				•	
Manganese		0	•			
Mercury	0		•			
Nickel		•		•		
Potassium	•		•			
Scandium			•			
Selenium	•					
Sodium	•		•			
Sultur						•
Thallium		•				
Thorium		•				
Uranium		•				
Vanadium	•		•			
Zinc <sup>.</sup>	•			•		

#### Analytical Methods

- A. Atomic Absorption Spectrometry
- B. Isotope Dilution Mass Spectrometry
- C. Neutron Activation Analysis
- D. Polarography
- E. D. C. Plasmas Atomic Emission Spectrometry
- F. Ion Chromatography

#### Analysts

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19. L. Kosta (Nuclear Chemistry Section, Josef Stefen Institute, Ljubljana, Yugoslavia

U. S. Department of Commerce Malcolm Baldrige Secretary

National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis

#### Standard Reference Material 1646

#### **Estuarine Sediment**

This Standard Reference Material is intended primarily for calibrating instrumentation and evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in sediments, and similar matrices.

<u>Values of Constituent Elements:</u> The certified values for the constituent elements are shown in Table 1. They are based on results obtained either by definitive methods or by two or more independent, reliable analytical methods. *Non-certified values*, which are given for information only, appear in Table 2. All values are based on a minimum sample size of 500 mg of the material dried as indicated under "Instructions for Drying".

#### Notice to Users:

Expiration of Certification: The certification of this SRM will be invalid 5 years after date of shipping.

<u>Use</u>: The material should be kept in its original bottle and shaken well before each use. A minimum sample of 500 mg of the dried material (see Instructions for Drying) should be used for any analytical determination to be related to a certified value of this certificate.

Statistical consultation was provided by K. R. Eberhardt of the Statistical Engineering Division.

The overall direction and coordination of the technical measurements leading to certification were performed in the Inorganic Analytical Research Division, E. L. Garner, Chief.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R. Alvarez.

Washington, D.C. 20234 June 7, 1982 (Revision of Certificate dated 1-6-82) George A. Uriano, Chief
Office of Standard Reference Materials

Table 1. Certified Concentration of Constituent Elements

Element	Concentration, weight %	Element	Concentration, weight %
Aluminum <sup>2b, c; 6</sup>	$6.25 \pm 0.20$	Magnesium <sup>1c;2c</sup>	$1.09 \pm 0.08$
Calcium <sup>2b,c;6</sup>	$0.83 \pm 0.03$	Phosphorus <sup>2a</sup> ;6	$0.054 \pm 0.005$
lron <sup>2c;4a;6</sup>	$3.35 \pm 0.10$		
	Concentration,		Concentration,
Element	µg/g	Element	<u>μ</u> g/g
Arsenic 1d;4b	11.6 ± 1.3	Manganese 1c;2c	375 $\pm 20$
Cadmium 1b, 3a, b; 4b	$0.36 \pm 0.07$	Mercury 1a;4b	$0.063 \pm 0.012$
Chromium <sup>1c</sup> ;3b;4a	76 ± 3	Nickel <sup>1b</sup> ; <sup>2c</sup> ; <sup>5</sup>	32 ± 3
Cobalt 1b; 4a	$10.5 \pm 1.3$	Vanadium <sup>2a,3a</sup>	94 ± 1
Copper 1c;2c;4b	18 ± 3	Zinc 1b, c; 2c; 3b; 5	138 ± 6
Lead 16;3a;5	$28.2 \pm 1.8$		
1. Atomic absorption spect	rometry	3. Isotope dilution	mass spectrometry
a. cold vapor		a. thermal ionizatio	n
b. graphite furnace		b. spark source	
c. flame		4. Neutron activation	
d. hydride generation		a. instrumental	
2. Atomic emission spectrometry		b. radiochemical	
a. dc plasma		5. Polarography	
b. flame		6. X-ray fluorescence s	pectrometry

b. flame
6. X-ray fluorescence spectrom
c. inductively coupled plasma

Notes: (1.) Analytical values are based on the "dry-weight" of material (see Instructions for Drying). Mercury should be determined on samples without drying and the results adjusted to a "dry-weight" basis by determining the moisture content of separate samples.

(2.) The estimated uncertainty for an element is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples 500 mg or more.

Table 2. Non-certified Concentrations of Constituent Elements

Note: The values shown in this table are not certified because they are not based on the results of either a definitive method or two or more independent analytical methods. These values are included, for information only, to provide additional information on the composition.

Element	Concentration, Weight %	Element	Concentration, Weight %
Potassium	(1.4)	Sulfur	(0.96)
Silicon	(31)	Titanium	(0.51)
Sodium	(2.0)		
	Concentration,		Concentration,
Element	μg/g	Element	μg/g
Antimony	(0.4)	Molybdenum	(2.0)
Beryllium	(1.5)	Rubidium	(87)
Cerium	(80)	Scandium	(10.8)
Cesium	(3.7)	Selenium	(0.6)
Europium	(1.5)	Tellurium	(0.5)
Germanium	(1.4)	Thallium	(0.5)
Lithium	(49)	Thorium	(10)

#### Analysts:

Inorganic Analytical Research Division, National Bureau of Standards. I. L. Barnes, M. B. Blackburn, C. G. Blundell, T. A. Butler, M. S. Epstein, T. E. Gills, J. W. Gramlich, R. R. Greenberg, S. Hanamura, W. R. Kelly, H. M. Kingston, L. Machlan, E. J. Maienthal, J. D. Messman, T. J. Murphy, T. C. Rains, T. A. Rush, R. Sedivy, and R. L. Watters, Jr.

#### Cooperating Analysts:

University of Tokyo, Tokyo, Japan; present address: Meteorological Research Institute; Tsukuba, Ibaraki, Japan; Y. Dokiya (NBS Guest Worker).

Division of Chemistry, National Research Council of Canada, Ottawa, Canada; S. Berman, A. Desaulniers, R. Sturgeon, A. Mykytuik, J. McLaren, V. Boyko, and P. Semeniuk.

Instructions for Drying: Except for mercury, elements should be determined on samples that have been dried at 110°C for 2 hours.

Mercury should be determined on undried samples. However, because the certified concentration is reported on a "dry-weight" basis, the concentration determined on undried samples should be adjusted for the moisture content of the samples.

Source and Preparation of Material: The material for this SRM was supplied by R. Huggett, Virginia Institute of Marine Sciences, Gloucester Point, Va. It had been dredged from the Chesapeake Bay at a location: 37° 11.1′ N, 76° 17.1′ W. The material was freeze-dried at Eastern Freeze-Dry Corporation, Lancaster, Pa., and radiation sterilized at Neutron Products Inc., Dickerson, Md. At NBS, the sediment was sieved through a screen with openings of 1.00 mm (No. 18) to remove coarse contaminants; ball-milled to pass a sieve with openings of 150 µm (No. 100); thoroughly mixed in a V-blender; placed in polyethylene bags; and bottled.

Homogeneity Assessment: A preliminary evaluation of homogeneity was made by instrumental neutron activation using samples of approximately 250 mg taken from various locations of the bulk materials. The samples were irradiated and the activities from radionuclides of Ce, Co, Cr, Cs, Eu, Fe, Rb, Sc and Th were counted. Except for Ce and Th, the observed sample-to-sample variations for the elements were approximately the same as the counting statistics indicating satisfactory homogeneity for these elements within approximately 2%. The homogeneity of the material for As, Cd, Hg, N, and Zn was evaluated by various analytical techniques using samples weighting 250 to 300 mg and found to be satisfactory. The homogeneity of the remaining certified elements was determined using sample weights not exceeding one gram.

The uncertainties of the elemental concentrations in Table I take into account possible material inhomogeneity for samples weighing 500 mg.

U. S. Department of Commerce Malcolm Baldrige Secretary National Bureau of Standards Ernest Ambler, Director

# National Bureau of Standards Certificate of Analysis

# Standard Reference Material 1648 Urban Particulate Matter

This Standard Reference Material (SRM) is intended for use in the calibration of apparatus and evaluation of methods used in the analysis of atmospheric particulate matter and materials with a similar matrix.

<u>Certified Values of Constituent Elements:</u> The certified values for the constituent elements are shown in Table 1. The analytical techniques used in the certification are shown in Table 3. The certified values are based on measurements of 6 to 30 samples by each of the analytical techniques indicated. Noncertified values are given for information only in Table 2.

Notice and Warnings to Users: This material is a naturally occuring urban dust to be used for analytical purposes only. It may contain a number of chemicals of unknown toxicities, therefore, the utmost caution and care must be exercised in its use.

Expiration of Certification: This certification is invalid after 5 years from date of purchase. Should it be shown to be invalid prior to that time, users will be notified by NBS.

Stability: This material should be kept in its original bottle and stored at temperatures between 10-30 °C. It should not be exposed to intense source of radiation, including ultraviolet lamps or sunlight. Ideally, the bottle should be kept in a desiccator at the recommended temperature.

<u>Use:</u> A minimum of 100 mg of the dried material (See Drying Instructions) should be used for any analytical determination to be related to the certified values of this certificate.

Source and Preparation of Material: This SRM was prepared from urban particulate matter collected in the St. Louis, Missouri, area in a baghouse specially designed for this purpose. The material was removed from the filter bags and combined in a single lot. This product was screened through a fine-mesh sieve to remove extraneous materials and thoroughly blended in a V-blender. The material was then bottled and sequentially numbered. The material was collected over a period in excess of 12 months and, therefore, is a time-integrated sample. While not represented to be typical of the area in which it was collected, its use should typify the analytical problems of atmospheric samples obtained from industrialized urban areas.

Homogeneity Assessment: Randomly selected bottles were used for the analytical measurements. Each analyst examined at least 6 bottles, some of them measuring replicates from each bottle. No correlation was found between measured values and the bottling sequence. Also, the results of measurements of samples from different bottles were not significantly different than the measurements of replicate samples from single bottles. Accordingly, all bottles of this SRM have been assigned the same certified values of constituent elements.

Instructions for Drying: This material should be dried at 105 °C for 8 hours before use. Because the certified concentrations are reported on a "dry-weight" basis, the concentrations determined on undried samples should be adjusted for the moisture content of the samples.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J.K. Taylor.

The technical and support aspects involved in the preparation, current and previous certification, and issuance of this Standard Reference Materials were coordinated through the Office of Standard Reference Materials by T.E. Gills and W.P. Reed.

Washington, D.C. 20234 May 11, 1982 (Revision of Certilicate dated 11-16-78) George A. Uriano, Chief Office of Standard Reference Materials

Table 1. Certified Values of Constituent Elements

Major Constituents		Minor Constituents		
	Content <sup>a</sup>		Content <sup>a</sup>	
Element	Wt. Percent	Element	Wt. Percent	
Aluminum <sup>b</sup>	$3.42 \pm 0.11$	Lead	$0.655 \pm 0.008$	
lron	$3.91 \pm 0.10$	Sodium <sup>b</sup>	$0.425 \pm 0.002$	
Potassium <sup>b</sup>	$1.05 \pm 0.01$	Zinc	$0.476 \pm 0.014$	

#### Trace Constituents

	Co	ntent <sup>a</sup>		Cor	ntent
Element	$\mu g/g$		Element	$\mu g/g$	
Arsenic	115	± 10	Nickel	82	± 3
Cadmium	75	± 7	Selenium <sup>b</sup>	27	± 1
Chromium	403	± 12	Uranium	5.5	$\pm 0.1$
Copper	609	$\pm$ 27	Vanadium b	140	$\pm 3$

The uncertainties shown for the elements except those noted by superscripts include errors associated with both measurement and material variability. They represent the 95 percent tolerance limits for individual subsamples, i.e., 95 percent of the subsamples from a single unit of this SRM would be expected to have a composition within the indicated range of values 95 percent of the time.

#### Table 2. Noncertified Values for Constituent Elements

Note: The following values are not certified because they are not based on the results of either a reference method or two or more independent methods. These values are included for information only.

Major Constituents		Minor Co	Minor Constituents			
Element	Content Wt. Percent	Element	Content Wt. Percent			
Sulfur	(5.0)	Chlorine	(0.45)			
Magnesium	(0.8)	Titanium	(0.40)			
	Trace	Constituents				
	Content		Content			
Element	$\mu g/g$	Element	<u>μg/g</u>			
Antimony	(45)	Lanthanum	(42)			
Barium	(737)	Rubidium	(52)			
Bromine	(500)	Manganese	(860)			
Cerium	(55)	Samarium	( 4.4)			
Cesium	(3)	Scandium	( 7)			
Cobalt	(18)	Silver	( 6)			
Europium	( 0.8)	Thorium	( 7.4)			
Hafnium	( 4.4)	Tungsten	( 4.8)			
Indium	( 1.0)					
lodine	(20)					

<sup>&</sup>lt;sup>b</sup>These elements were recently certified as a part of the NBS update certification program. The values for the indicated constituent are the "best value" based on all measurement methods used and the associated uncertainty is expressed as the standard error considering variability within and between analytical methods.

#### Supplemental Information

The values listed below are based on measurements made in a single laboratory and are given for information only. While no reason exists to suspect systematic bias in these numbers, no attempt was made to evaluate such bias attributable to either the method or the laboratory. The method used for each set of measurements is also listed. The uncertainties indicated are two times the standard deviation of the means.

Constituent	Content Wt. Percent
Nitrogen (NO <sub>3</sub> )	$(1.07 \pm 0.06)$
Nitrogen (NH4)	$(2.01 \pm 0.08)$
Sulfate	$(15.42 \pm 0.14)$
$SiO_2$	$(26.8 \pm 0.38)$
Ficon Soluble	$(1.19 \pm 0.47)$

#### Methods Used:

Nitrate - Extraction with water and measurement by ASTM Method D992

Ammonia - NaOH addition followed by steam distillation and titration

Sulfate - Extraction with water and measurement by ASTM D516

SiO<sub>2</sub> - Solution and measurement by ASTM Method E350

Freon Soluble - Extraction with Freon 113, using the Method described in "Standard Methods in Examination of Water and Waste Water," 14th Edition, p. 518, American Public Health Association, Washington, D.C.

#### Analysts

Inorganic Analytical Chemistry Division

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7. M. Gallorini	15. T. C. Rains
8. E. L. Garner	16. T. A. Rush

Table 3 Methods and Analysis

Method/ Element	А	В	С	D	Е	F	G	Н	ı
Ag			•						
Al			•					•	
As			•		•				
Ва			•						
Br			•						
Cd	•	•	•	•					
Се			•						
CI							•		
Со			•						
Cr		•	•						
Cs			•						
Cu	•	•			•				
Eu			•						
Fe	•		•		•				
Hf			•						
I			•			•			
ln			•						
K	•		•						
La									
Mg			•						
Mn	•		•						•
Na	•	-	•						•
Ni	•	•		•					
Pb	•	•		•					
Rb			•						
S							•		
Sb			•						
Sc			•						
Se	•		•						•
Sm			•						
Th			•						
Ti			•						
U		•							
V	•		•						
W			•						
Zn	•	•	•	•					

#### Analytical Methods

- A. Atomic Absorption Spectrometry
- B. Isotope Dilution Mass Spectrometry
- C. Neutron Activation Analysis
- D. Polarography
- E. Spectrophotometry
- F. Photon Activation Analysis
- G. Ion Chromatography
- H. D.C. Plasma Atomic Emission Spectrometry
- 1. Flame Emission Spectrometry

# Guide for Requesting Development of Standard Reference Materials

The National Bureau of Standards develops Standard Reference Materials (SRM's) to provide a basis for comparison of measurements on materials and to aid in the control of production processes. The Office of Standard Reference Materials evaluates the requirements of science, industry, and government for carefully characterized reference materials, then directs the production and distribution of these materials.

NBS currently has over 1000 SRM's available, about 100 new ones in preparation, and requests for the development of many more. The demand for new SRM's greatly exceeds the Bureau's capacity to produce and certify these materials. Consequently, requests for new SRM's of limited use are deferred in favor of those that serve a substantial area of interest. In determining which requests receive top priority, NBS relies heavily upon information supplied by industry and interested organizations.

The Bureau welcomes all requests for SRM's. Both the Bureau and potential users would be helped if these requests included as much of the information below as possible.

- 1. Short title of the proposed Standard Reference Material.
- 2. Purpose for which this SRM is intended.
- 3. Reason why the SRM would be useful.
- **4.** Special characteristics and/or requirements of the material. Include necessary additional information, if more than one SRM is needed for standardization in an area.
- **5.** An estimate of the possible present and future (6-10 years) demand for such an SRM in your operations and elsewhere. (National and international estimates are very useful).

- **6.** Facts about whether such an SRM (or a similar one) could be produced by, or obtained from a source other than NBS. If so, justify preparation by NRS
- **7.** Other pertinent information to justify the SRM, such as: (a) an estimate of the range of application, economic significance of the measurement affected, and scientific and/or technological significance, including estimates of the impact upon industrial productivity or growth, and (b) supporting letters from industry leaders, trade organizations, interested committees, and others.

In developing an NBS-SRM, the candidate material must r set one or more of the criteria listed below:

- **1.** The SRM must permit users to attain more accurate measurements.
- **2.** The production of the SRM must not be economically or technically feasible elsewhere,
- **3.** The SRM must serve as an industry-wide standard for commerce, provided by a unique neutral source,
- **4.** NBS production of the SRM would provide readily available, highly characterized material useful to science, industry, or government.

NBS has recognized the need to enlarge the scope of the SRM program to include all types of well-characterized materials that can be used to calibrate a measurement system, or to produce scientific data that can be widely used. Input from science, industry, and government assists NBS in continuing to provide Standard Reference Materials that will be valuable in many areas.

#### GUIDE TO ORDERING STANDARD REFERENCE MATERIALS

#### ORDERING

Orders should be addressed to:
Office of Standard Reference Materials
Room B311, Chemistry Building
National Bureau of Standards
Gaithersburg, MD 20899
Telephone: (301) 921-2045

Orders should give number of units, catalog number, and name of the material requested. For example, I each, No. 11h, Basic-Open-Hearth Steel, 0.2 percent C. The materials described in this Catalog are distributed only in the units listed or in multiples thereof.

Acceptance of an order does not imply acceptance of any provision set forth in the order contrary to the policy, practice, or regulations of the National Bureau of Standards or the U.S. Government.

Orders received for "out-of-stock" materials are cancelled if only out-of-stock items are ordered. On other orders, shipment is made of available materials and out-of-stock items are cancelled. Back-orders are not accepted for out-of-stock materials; if a renewal lot of material is available, it will be furnished automatically.

#### TERMS

Prices quoted are in U.S. dollars, and are published in the SRM Price List. When SRM Price Lists are issued they are sent to persons or organizations who have requested them. These prices are subject to revision without notice and orders will be billed for the prices in effect at the time of shipment. No discounts are given on purchases of SRM's, RM's, or GM's.

Remittances of the purchase price need not accompany purchase orders. Payment of invoices is expected within 30 days of receipt of an invoice. Payment on foreign orders may be made by any of the following:

- a. banker's draft against U.S.A. bank
- b. bank to bank transfer to U.S.A. bank
- c. cash against documents
- d. sight draft
- e. International Money Order
- f. UNESCO coupons

Letters of Credit cannot be accepted. If a Letter of Credit or any method of payment other than those listed above is to be used, you must secure the services of an agent in the United States to act in your behalf. Your agent would purchase the material and our invoice would indicate that he is the purchaser. The material would be shipped to your agent, who would transship in accordance with your instructions.

NBS cannot "prepay and add" shipping charges to the invoice. Restricted categories such as hydrocarbons, organic sulfur compounds, compressed gasses, rubber compounding materials, radioactive standards, and similar materials are shipped FOB Gaithersburg, MD.

#### LATE CHARGES

Unless otherwise notified, payment for SRM's is due within 30 days of shipment of the order to the customer. For non-Federal customers, the U.S. Treasury regulations require late charges, based on the current value of funds to Treasury, be assessed for each 30-day period or portion thereof that the payment is overdue.

#### PROFORMA INVOICE (PRICE QUOTATION)

Proforma invoice service will frequently require three to four weeks to process, and will be furnished only to those requiring such service.

#### DOMESTIC SHIPMENTS

Shipments of material (except for certain restricted categories) intended for the United States and Canada are normally shipped prepaid (providing that the proel does not exceed the weight limitations as prescribed by postal laws and regulations).

#### FOREIGN SHIPMENTS

The regulations of various nations covering the importation of SRM's, GM's, and RM's differ widely; any attempt to list all possible variations would be impractical. Therefore, where the shipping practices outlined below do not apply, purchasers will be informed of the best method of shipment for their countries.

Most orders will be shipped by prepaid International Air Parcel Post. Exceptions are items in restricted categories and those shipments that exceed parcel post weight limitations. These exceptions will be shipped FOB Gaithersburg, MD, unless an agent (shipping or brokerage firm) located in the United States is required. Where an agent is required, the purchaser will be so notified and will be requested to designate an agent of his/her choice. In this case, the material will be packaged for overseas shipment and will be forwarded to the agent FOB Gaithersburg, MD.

#### DOCUMENTATION

Listed below are the only documents that we will furnish. All documents are printed in English.

- a. six commercial invoices
- b. two sight drafts
- c. two packing slips
- d. customs invoces for Canada, New Zealand, Australia, and South Africa
- e. Certificate of Origin
- f. parcel post receipts for parcel post shipments
- g. air waybill for air shipments

If documents other than those listed above are required, the services of an agent in the United States will be needed to purchase and ship the materials.

Note: Orders and inquiries submitted in English will be processed more rapidly than those requiring translations.

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NBS-114A (REV. 2-8C)						
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Tibrary of Congres	ss Catalog Card No: 8	06-600504				
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		S Software Summary, is attached.				
<ol> <li>ABSTRACT (A 200-word of bibliography or literature)</li> </ol>	r less factual summary of most survey, mention it here)	significant information. If documer	nt includes a significant			
This publication	is a summary of the e	nvironmental research,	analysis. and			
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